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TEXTILE RESEARCH JOURNAL

VOLUME XVIII | October, 1948 | NUMBER 10

PUBLICATION OF

TEXTILE RESEARCH INSTITUTE, INC.

AND

THE TEXTILE FOUNDATION

Editorial Office: 10 East 40th Street, New York 16, N. Y.

TEXTILE RESEARCH JOURNAL

VOL. XVIII, No. 10 October, 1948

In This Issue

Radiation Drying of Textiles
George T. Paul and Richard H. Wilhelm 573

Dyeing Studies at Elevated Temperatures
Use of Temperatures Between 200° F and 300° F
G. L. Royer, C. L. Zimmerman, H. J. Walter, and R. D. Robinson 598

A Laboratory Study of Padding
Part I—Cotton
C. L. Zimmerman and G. L. Royer 615

Degree of Polymerization of Cellulose in Cotton Fibers
Lyle E. Hessler, George V. Merola, and Earl E. Berkley 628

Discussion of Published Papers
Remarks on the "Secondary Yield Point" in Wet Wool Fibers
Harris M. Burte 635

Abstracts 636

Exhibitors at T. R. I. Annual Meeting i

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TEXTILE RESEARCH JOURNAL, published monthly at Prince and Lemon Streets, Lancaster, Pa., is devoted to the interest of research in textile and allied industries. It is the official publication of Textile Research Institute, Inc., and the Textile Foundation.

Manuscripts and communications should be sent to the editorial offices, 10 East 40th Street, New York 16, N. Y.

Subscription rates: General—$15.00 per year; additional for postage, 50 cents to Canada, $1.00 to all other countries. Special—$10.00 per year for United States and Canadian students, teachers, educational institutions, public libraries, governmental libraries and departments, plus 50 cents for postage to Canada. Single copies, $1.50 each.

Entered as second-class matter, April 27, 1945, at the postoffice at Lancaster, Pa., under the Act of August 24, 1912.

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TEXTILE RESEARCH JOURNAL

VOL. XVIII, No. 10 October, 1948

Radiation Drying of Textiles

George T. Paul and Richard H. Wilhelm

Contribution of the Textile Research Institute and of the Department of Chemical Engineering, Princeton University, Princeton, New Jersey

Abstract

This paper deals with radiation drying of woven and matted wool felts, of woven wool crepe, and of absorbent cotton batting. Radiant energy was supplied by R–40 electric lamps, and air conditions at the surface of the samples were controlled. Drying rate curves are analyzed in terms of drying mechanisms, and comparisons are made with convection drying by means of heated air. Among the variables discussed are air conditions, air velocity, intensity of radiant energy, sample thickness, optical properties of the materials being dried, and the initial regain.

AS A MEANS of transmitting heat energy, radiation has certain attractive features. It is applied by direct, linear propagation and involves no intermediate heat storage; it lends itself to instantaneous control; and because of the high energy flow rates that may be achieved, equipment sizes may be correspondingly small and process time cycles may be short. Based upon these features, an important infrared heating industry has been developed within the last decade. Heating, to raise the temperature of materials, baking or curing, to cause a chemical reaction to take place, and drying, to remove water or organic solvents, are industrial operations being performed. Typical examples are baking of resins in surface coatings and in textile finishes, softening thermoplastics, shrink-fitting metal parts, softening adhesives, heat-set printing and drying plaster and clay molds, electrical insulation, wood pulp products, and textile materials.

Radiation drying of textile materials by means of high-intensity sources such as heat lamps and radiant gas burners, which is the specific subject of this paper, was introduced into the textile industry at about the beginning of World War II. To date, radiation driers have been used mainly to increase the capacity of existing drying equipment such as loop driers and drying cans [7]. Near infrared units are well suited for such booster service because they

may be built to fit within a relatively small floor area. It is natural, however, that questions should be raised concerning a new technique, such as radiation drying, which is in an early state of development. Such questions can be divided into two categories. The first relates to the mechanism and rate of drying of textiles as these depend upon variables such as radiation intensity, air conditions, and textile properties. The second question relates to the design of driers and deals with such problems as the selection and spacing of the radiant heat source, reflector design, and with provisions for controlled air flow. The first point of interest is what happens to a material being dried in a given environment, and this may be called "drying theory"; and the second relates to the most efficient means of providing such an environment. The latter is the province of design engineering. The two are interrelated in determining the optimum balance between operating cost and equipment cost. This paper will deal only with aspects of the first question; *no indication of economic advantage of any method of energy application, radiation or convection drying, should be construed from apparent scientific advantages without consideration of variables such as insulation, size of oven, moisture transport, and exhaust or conveying means which have a great bearing upon the efficiency of manufacturing equipment. The latter items are not considered in the present paper.* Specifically, the objectives of this paper are:

1. To compare the mechanisms for moisture removal in radiation drying with those in ordinary convection air drying.

2. To compare the rates in radiation and convection drying and the manner in which these are affected by the independent variables: radiant intensity, air flow rate, air temperature and humidity, and certain sample properties.

The fundamental literature on infrared radiation heating and drying is quite limited. Tiller and Garber [19] in a pioneer article presented theoretical derivations for time-temperature expressions for the radiation heating of thin metallic bodies. Ernst and Schumacher [6] compared the curing behavior of resin finishes on steel sheets baked by radiant heat and by convection heat and found no significant difference between the two at equal rates of heat flow. A comparison of mechanism and rate of drying by near infrared radiation with that for air drying was made by Stout, Caplan, and Baird [18]. Sand, soap, and magnesium stearates are the materials which were dried. The broad conclusions of the present paper regarding drying mechanisms agree with those of Stout, Caplan, and Baird, but radiant penetration apparently is greater in the textile materials here studied than in the solids listed above. Infrared drying of textiles has recently been studied by Preston and Chen [16] with emphasis on determination of the second critical regain by means of the temperature, and on the absorptivity of the fabric. Ickis and Haynes [10] presented a review of the characteristics of radiant energy and of drying lamps. The production, transmission, reflection, and measurement of infrared radiation were the subjects of a review by Koller [13]. Definitions of terms, the laws of radiant emission, absorption, reflection, and the measurement and analysis of radiant energy have been reviewed by a committee on colorimetry of the Optical Society of America [5].

Experimental Procedure

Apparatus

The radiation drier consisted of two opposed horizontal banks of lamps. Each bank contained 7 lamps

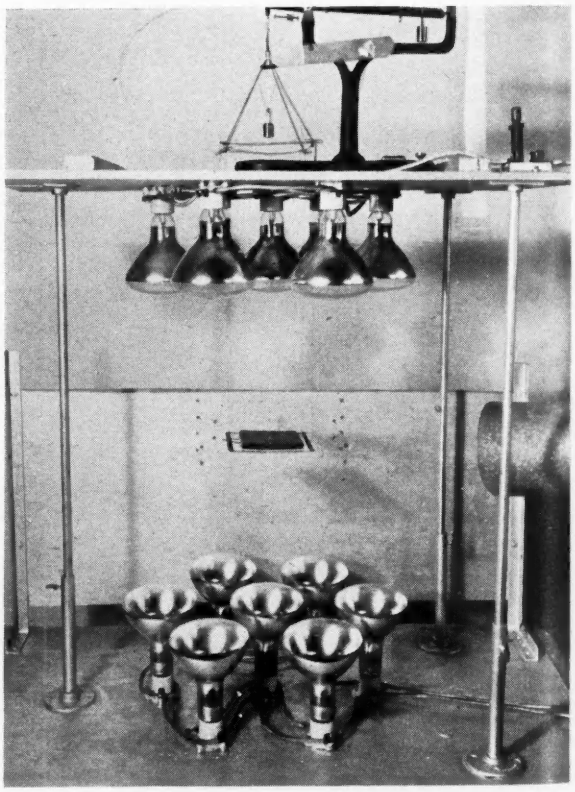

FIG. 1. *Radiation drying equipment with electric heating lamps.*

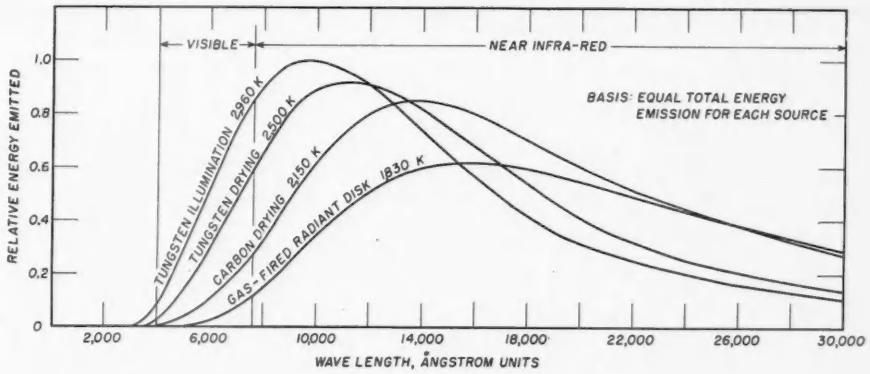

FIG. 2. *Spectral distribution of radiant energy sources.*

in hexagonal arrangement, 7 inches from center to center. A frame for holding the sample to be dried was suspended from a balance above the apparatus and was held horizontally in a median position between the lamp banks. This is shown in Figure 1. The frame was made of aluminum, and the suspensions, as well as the lacing on the frame, were made of Saran filament, which absorbs no water. The balance, with which the change in weight of the sample was measured as drying proceeded, was a Will balance with a total load of 250 grams and a sensitivity of 0.005 gram.

R-40 lamps were used as the source of infrared radiation. These are 250-watt internal-reflector, tungsten-filament lamps operating at a temperature of 2,500°K. The spectral distribution is given in Figure 2. A comparison is also presented in this figure between these 2,500°K tungsten drying lamps and 2,960°K tungsten illumination lamps, carbon-filament drying lamps at 2,150°K, and a radiant source at 1,830°K, the temperature at which one gas-fired type of radiant disc has been stated to operate. All curves are plotted for equal total illumination summed over all wave lengths.

Provision was made for an air stream to blow over the sample parallel to the surface. Velocities were controlled between 0 and 4 meters per second (0–13 feet per second). The open end of the air duct is visible at the right in Figure 1. Aside from a flattening, with the wide axis horizontal, the duct was built to standard specifications [15] and contained a straightener and a calming section. Traverses of the air flow stream at the position of the sample with a calibrated anemometer showed the stream to be uniform within 5 percent over the sample width.

The distance between lamp banks was variable and permitted a considerable control in radiation intensity at the work surface. R-40 lamps do not give a perfectly uniform illumination pattern, but tend to show an image of the tungsten filament. More significant, the angular distribution of energy differs appreciably from lamp to lamp, leading to different flux intensities on the line of the axis and on the sample. In continuous industrial drying, owing to the relative motion between stock and lamps, this variation is of little importance because average intensities at all points on the stock are nearly equal. For the purposes of the present type of experiment, however, to secure within a few percent a uniform field of illumination in an area about 125 square centimeters, it was necessary to make measurements on the illumination pattern on a plane perpendicular to the lamp axes and then to match lamps carefully in assembling them in a bank.

Sample temperatures during a run were determined by means of thermocouples inserted close to the upper and lower surfaces and in the middle of the cross section. In Figure 1 thermocouple wires may be seen leading through holes in the aluminum-painted back board of the drier. From these, connections were made to ice-bath cold junctions and to a Leeds and Northrup portable potentiometer-indicator model 8657C. The thermocouples consisted of No. 28 copper and No. 32 constantan wires, silver-soldered at both hot and cold junctions. In forming the junctions, excess molten solder was shaken off to yield small, clean joints which would not deform the sample. The wires were insulated with glyptal resin containing aluminum flakes to minimize absorption by the wires of stray radiant energy from the lamps.

In air-drying experiments a laboratory model Sturtevant cabinet drier, steam-heated, with provision for steam injection for humidity control, was used. A long approach pipe and straightening vanes were installed to give a uniform air flow pattern at the position of the sample. Air velocity was varied

through use of orifices in the recirculation line. The maximum temperature attainable was limited by steam pressure and air velocity; hence, the range of air-drying experiments was limited. Weight decrements during drying were measured by the same type of balance used in the radiation equipment. The manner of sample suspension and thermocouple attachment also were similar.

Three different instruments used for the measurement of radiant intensity are shown in Figure 3. From left to right, the first is an 8-junction Eppley thermopile with sodium chloride window, Model No. 1038; the second is a General Electric Model ZA-4, radiation-type, single-junction vacuum thermocouple; and the third is a 10-junction Eppley pyrheliometer. All three units are of the thermopile type with a blackened surface radiation receiver and, as a consequence, they have the advantage of equal adsorption in all wave lengths of radiant energy. The Eppley thermopile was used in calibrating and exploring the radiation from individual drying lamps. However, because of a built-in angle of reception in the metal case, the unit was not suitable for measuring the radiant energy from a bank of lamps. The vacuum thermocouple was found useful in exploratory work, but because of a non-linear calibration and because of a tendency to overheat rapidly, this tube was not found suitable for measuring absolute values of radiant-field intensities. The pyrheliometer proved to be most suitably adapted to this latter purpose of any instrument found commercially, because of a flat construction which permits the sighting and measurement of energy through an azimuth of 180°.

The method of adjusting and judging the uniformity of radiant energy from each bank of lamps was simple, direct, and rapid, and may be useful for this purpose industrially. It depends upon the property of cobalt chloride of changing from a pink to a blue color as it is taken from a wet to a dry state. The color change is the result of variations in coordination complexes between water and cobalt chloride. A white cotton cloth was immersed in a 20-percent solution of cobalt chloride hexahydrate and was run between rollers to achieve as uniform a distribution of the cobalt chloride solution as possible. The pink-colored cloth was thereupon stretched upon a small tenter frame and placed in the radiation to be studied with the lamp axis perpendicular to the cloth. In a uniform field of radiation, drying proceeded at an equal rate at all points and a uniform change from pink to blue

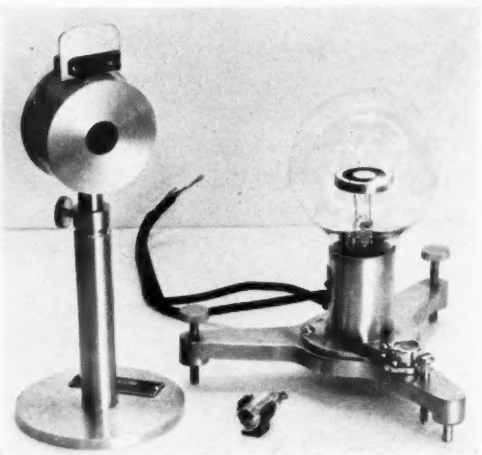

FIG. 3. *Thermopile, vacuum thermocouple, and pyrheliometer for measuring radiant energy.*

occurred. Any differences in intensity resulted in the formation of easily distinguishable patterns of blue on a pink background, which could be photographed, be referred to cross-section lines on the cloth, or otherwise noted. Depending upon humidity conditions in the room, the patterns were retained as long as 15 minutes. The method was much more rapid and generally satisfactory compared to exploring with a thermopile or vacuum thermocouple. As background for this method, equilibrium measurements were made on the system: cotton, water, cobalt chloride. Using a 20-percent cobalt chloride solution and an initial regain of 67 percent, the distinguishable change from pink to blue took place between regains of 10 and 7.5 percent. However, great care must be exercised in any attempt to relate color change with change in moisture content because strong evidence has been obtained that cobalt chloride also forms coordination complexes with cotton as well as with water and the color depends on the cobalt chloride content of the cloth as well as on the regain.

Materials

Materials used in this study were wool and cotton, primarily the former. Specifications are as follows:

1. Woven wool felt
   Color: light brown
   Thicknesses: 0.95 cm. (0.35 inch)
   1.4 cm. (0.55 inch)
2. Matted wool felt
   Color: gray
   S.A.E. specification F-3

Thicknesses: 0.19 cm. (0.076 inch)
0.64 cm. (0.25 inch)
2.0 cm. (0.78 inch)

3. Matted wool felt
   Color: white
   S.A.E. specification F-10
   Thickness: 0.66 cm. (0.26 inch)
4. Woven wool crepe
   Color: white
   Thickness: 0.084 cm. (0.033 inch)
5. Absorbent cotton batting
   Color: white
   Thickness, dry 4 cm. (1.6 inch)
   wet 0.4 cm. (0.16 inch)

Some samples of the white wools, types 3 and 4, were dyed with Wool Black 4B (Kahlbaum), and some with a black mixture containing Calcocid Green B.

These materials, predominantly thick, were used purposely because they furnished opportunity for study of such effects as radiant penetration through incorporation of thermocouples and because experiments with such materials were of sufficiently long duration to permit adequate precision.

Procedure

Sample Preparation. The material was cut into squares of approximately 10 centimeters on a side. Using a soldering iron with temperature controlled by a variable transformer, each sample was coated on the four edges with sealing wax to prevent evaporation of water and to minimize heat transmission through the edges. The wax weight was obtained by weighing the stick of sealing wax and the soldering iron tip before and after waxing. The sample was dried successively in two ovens at 110°C, using calcium sulfate desiccant in the second oven. The constant weight obtained after several days was considered to be dry weight.

Depending on the sample thickness, one or three holes were drilled in one edge for thermocouple insertion. The initial puncture was made with a No. 50 wire drill, and this was deepened with a sharpened smooth wire 0.94 millimeter in diameter, a length of which was left in the puncture to prevent its collapse. The depths of all wires beneath the surfaces were measured by means of a needle probe and scale to the nearest 0.25 millimeter. In a typical 0.64-centimeter sample, the surface thermocouple junctions were located at a depth of 0.08 centimeter and the center thermocouple junction 0.28 centimeter from one surface and 0.36 centimeter from the other. Depth tolerances employed for the surface junctions were smaller than for the center one because of the possibilities for steeper temperature gradients at the surface and because of the necessity of preventing the hot junctions from being "seen" by the radiation source.

For theoretical reasons most samples were wetted thoroughly by the procedure which is here described. As is discussed later, some samples were centrifuged to obtain initial regains comparable with those used in industrial practice. In normal practice in this work, therefore, the sample was thoroughly wetted with distilled water by submersion in a water-filled desiccator in which atmospheric pressure was alternately removed and reapplied by an aspirator and three-way valve. After drainage of excess water, the sample was transferred to the sample support in the drier. The wire rods in the thermocouple wells were removed and the hot junctions were inserted to depths of 2.5 to 4 centimeters from the edge. Sample surfaces were gently tamped by hand to insure good contact between fibers and hot junctions. The restraining Saran threads were then used to clamp the sample rigidly on the holder despite the air stream.

The sample was thoroughly rewetted by pouring water onto all parts of both surfaces until no dry surface areas remained. Air-stream control was established by the use of the appropriate orifice in the air intake. Initial temperature measurements were obtained on all thermocouples. The sample was weighed with and without the air stream blowing across it. To conclude preparations, the balance was adjusted to the integral weight reading directly below that of the actual sample weight.

Typical Run. The heating lamps and the air blower with the proper orifice in place were turned on, and zero time was defined when the sample weight passed through the integral reading on the balance mentioned above. Weights were noted by the observer at regular weight intervals and recorded, together with the time at which they occurred, by an assistant. The intervals used initially were 2 grams, but were decreased gradually to 0.2 or 0.3 gram as dry weight was approached. Thermocouple readings were taken initially at weight intervals of 4 grams, but as the temperature rose, these were noted as frequently as possible. Visual observations of sample condition were made at convenient intervals. When

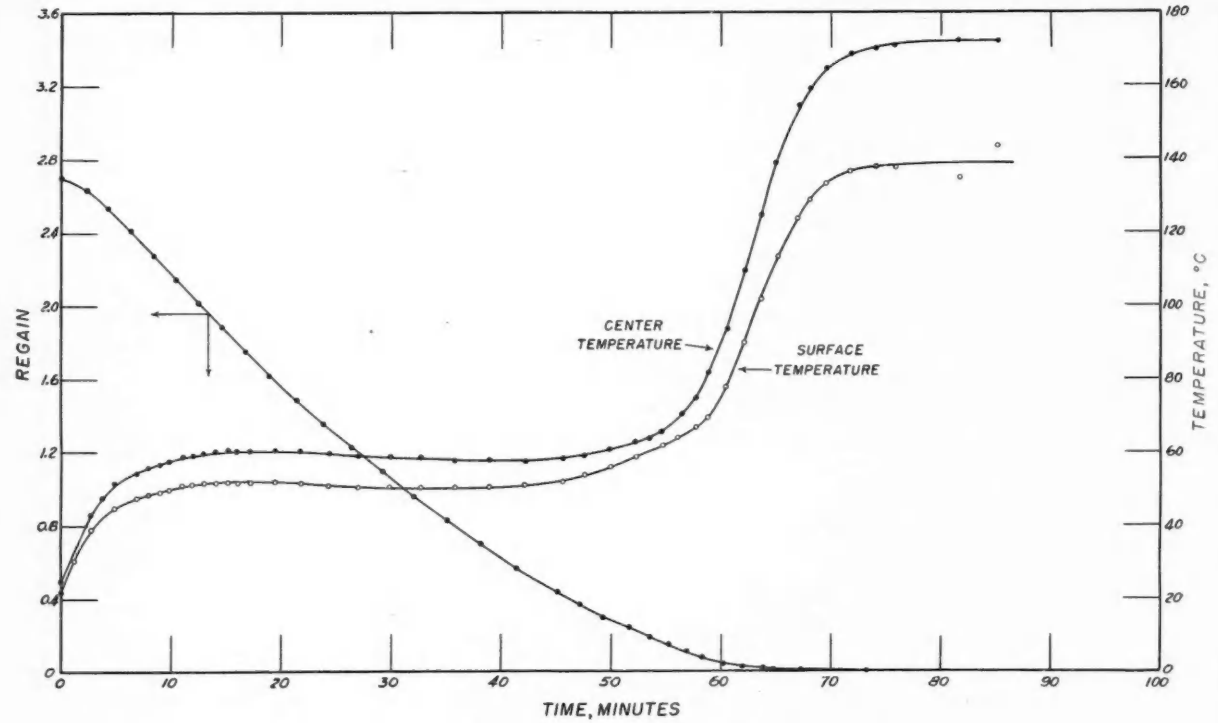

FIG. 4. *Regain and temperature versus time. Radiation drying of woven wool felt.*

no further loss in weight could be observed and temperature had become constant, the run was complete.

At the end of a run, sample weight was measured with and without the air stream. Then, after switching off the lamps and blower, weighings were made with the center thermocouple in place as the sample was allowed to cool in stagnant air. A final weighing was made after removal of the thermocouple. Wet- and dry-bulb temperatures were measured at the beginning and end of each run.

Treatment of Data. Primary data obtained were apparent weight of sample and thermocouple potentiometer readings at known time intervals. The apparent weight was corrected for the effects of thermocouple wires, sample temperature (a buoyancy effect), the vertical component of the air stream thrust, and the weight of the sealing wax. Potentiometer millivolt readings were converted to temperatures, using a calibration curve determined with a thermometer calibrated by the U. S. Bureau of Standards. Processed data thus obtained were values of regain (in weight moisture per weight

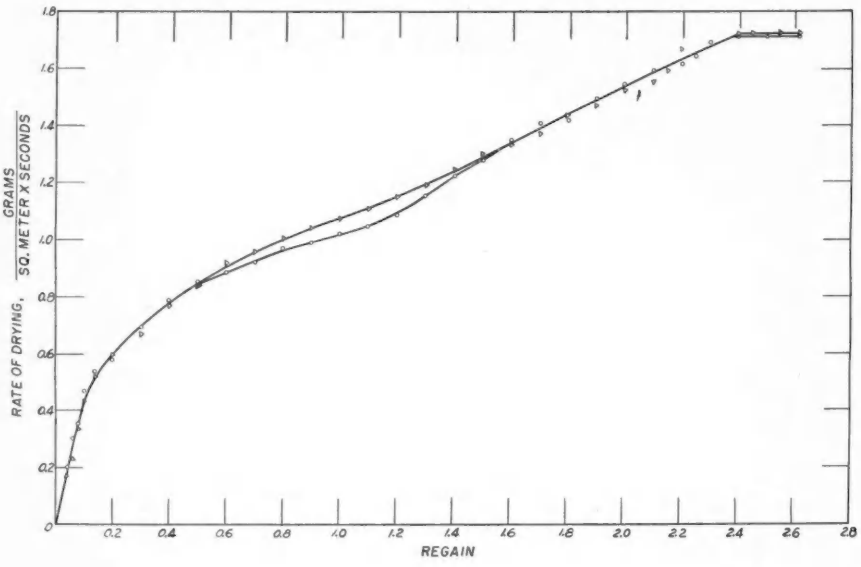

FIG. 5. *Rate of drying versus regain. Radiation drying of woven wool felt.*

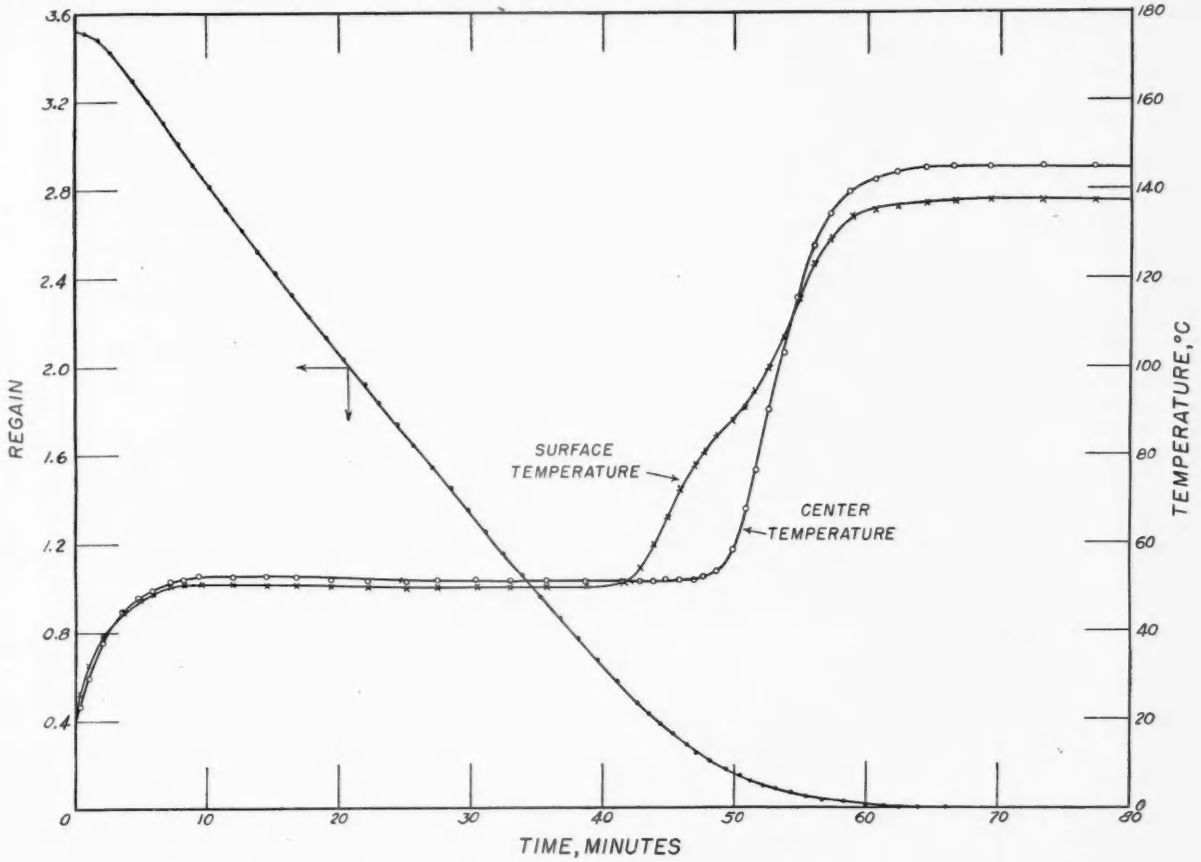

FIG. 6. *Regain and temperature versus time. Radiation drying of matted wool felt.*

dry solids) versus time, and temperature versus time. Figures 4 and 6 provide examples of such data.

The usual and most profitable manner of presenting drying data * is in the form of drying-rate-versus-regain curves. All data were computed to this basis. Rate curves were prepared through tangents, or first derivatives, of the regain-versus-time curves and, from these tangents, the rate of drying was computed. The latter was expressed in grams of water removed per square meter per second. Tangents in this work were measured by several persons and were reproducible within 0.5 percent. Typical rate-regain curves are given in Figures 5, 7, and 9.

Figure 5 demonstrates the reproducibility in results attained in drying the same sample twice under equivalent drying conditions. The curves differ slightly only during a portion of the first falling rate period, a behavior which is not unlikely because of a lack of complete reproducibility of capillary flow conditions.

Figure 6 shows the temperature relations which were exhibited in most of the runs. Starting at the same temperature before the heating lamps were turned on, the surface of the sample heated more rapidly than the center, but for a comparatively long, steady-state period during the drying operation, and again after drying had been completed, the center temperature reached a value higher than that of the surface. This is taken as direct evidence of transmission of radiant energy into the felt.

Because of small sample size, drying rates in this project may be considered to have been measured within any one run at essentially constant conditions of air temperature, air humidity, and radiation intensity. In an industrial drier the temperature and humidity of the air stream vary from point to point within the drier, and the material is subjected to a changing environment as it proceeds from entrance

* The original time-regain data and curves upon which rate-regain graphs are based are available for inspection and for copying in the laboratory of the Textile Research Institute, Princeton, N. J.

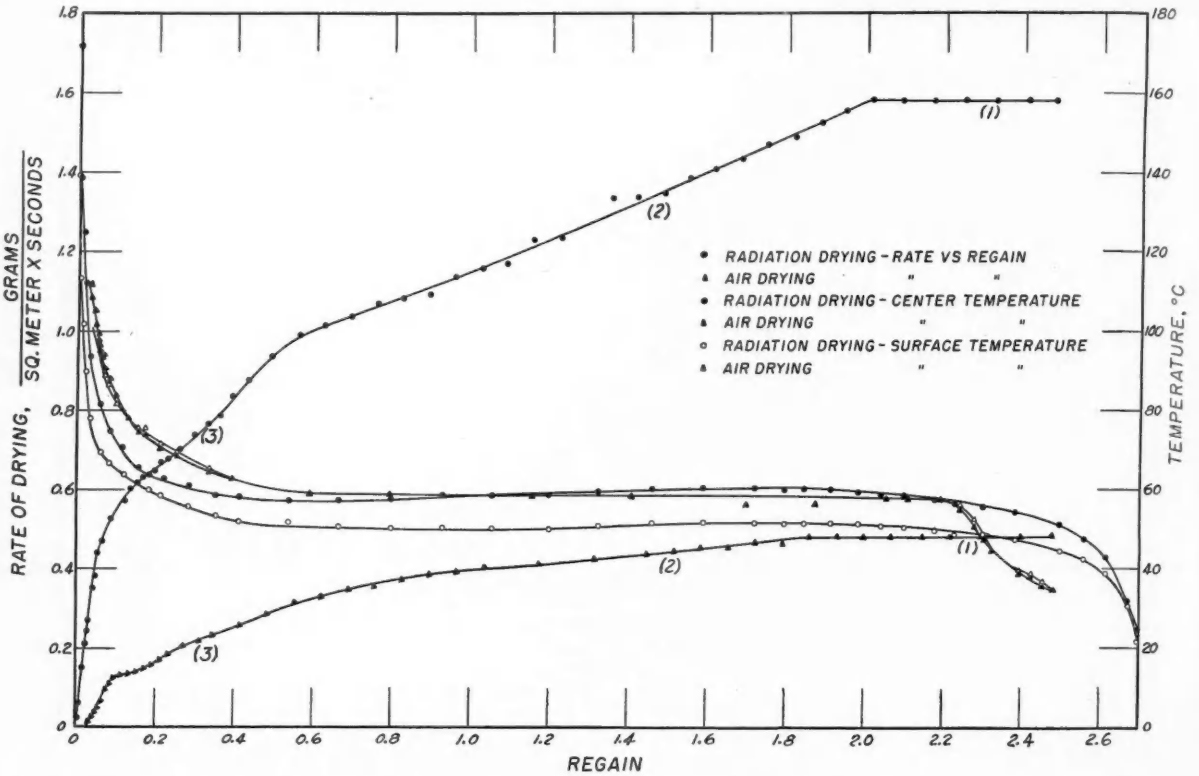

FIG. 7. *Rate of drying and temperature versus regain. Radiation drying and air drying of woven wool felt.*

to exit. The rate of drying will be affected by these changing conditions. To design a drier with respect to capacity, radiation input, air-flow quantity and conditions, and time of drying, there are required (1) experimental rate-regain information, such as is presented in this paper, concerning the rates of drying as they are affected by conditions; and (2) heat, material, and water balances. Procedures and examples of drier design from rate data are available in the literature [8, 21].

Mechanism of Drying

In studying the drying of textiles by radiant energy, it is of interest to consider mechanisms for the removal of moisture from such materials and to compare these between radiation drying and ordinary convection drying with heated air. By a textile is meant the bulk structure that is formed by the weaving, knitting, or matting of textile fibers. The fact that the fibers are in a bulk structure, permeated with air, contributes to the drying characteristics and causes differences in the sequence of events as compared, for example, with the drying of individual fibers.

A suitable and commonly used basis for analyzing drying mechanisms is the experimental rate-regain curve. Such curves for 0.95-centimeter woven wool felt, dried both by air and by radiant energy, are presented in Figure 7. Three periods of drying, appropriately numbered, are evident in each of these curves. The three associated mechanisms are presented diagrammatically in Figure 8. The first period of moisture removal is characterized by a constant rate of drying and is evident only when excess free surface water is present. Although not commonly encountered in textile drying practice,

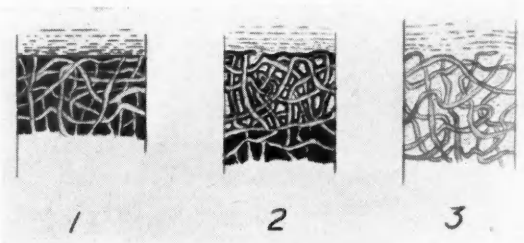

FIG. 8. *Diagrammatic representation of felt showing relation between fiber and moisture in various periods of drying.*

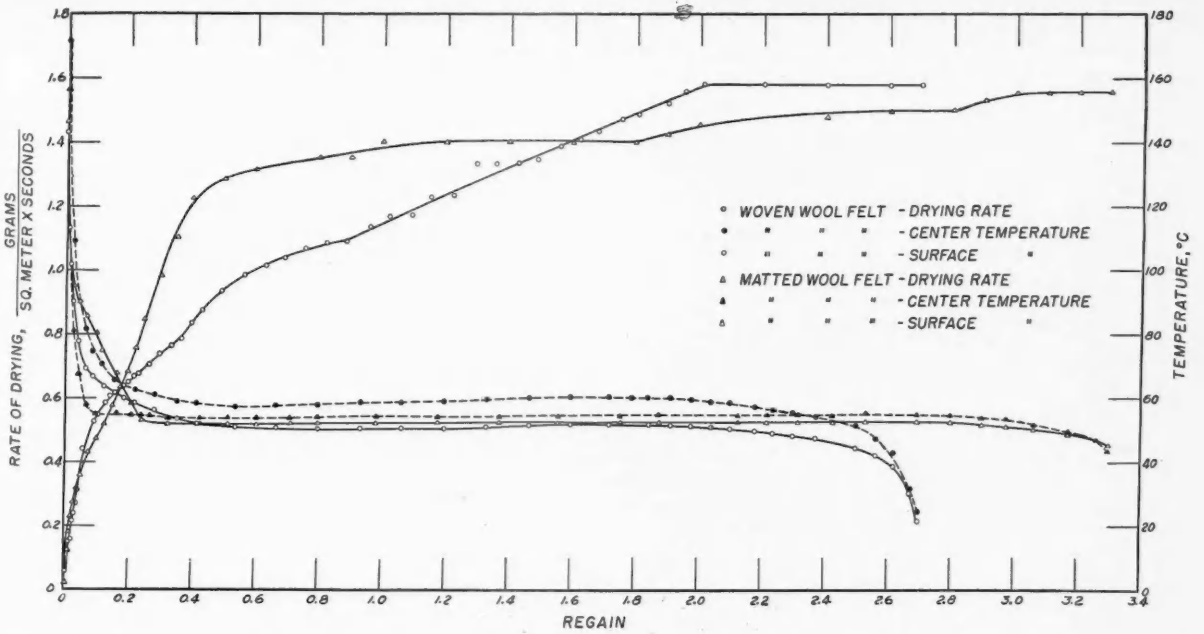
RATE OF DRYING, GRAMS / SQ. METER X SECONDS
TEMPERATURE, °C
REGAIN
WOVEN WOOL FELT - DRYING RATE
" " " - CENTER TEMPERATURE
" " " - SURFACE "
MATTED WOOL FELT - DRYING RATE
" " " - CENTER TEMPERATURE
" " " - SURFACE "

FIG. 9. *Rate of drying and temperature versus regain. Radiation drying of woven and matted wool felts.*

this period was included in most experiments by starting with thoroughly wetted material, because, among the drying periods, this is the most susceptible to analysis. The second period, a falling-rate period, involves the capillary migration of water to the surface zone, where it is evaporated, a mechanism that is accompanied by the penetration of air into spaces between the fibers. The third and final period represents the removal of water by diffusion from within the individual fibers of the felt and the migration by diffusion of the vapor to the surface. Appropriately, this is called the diffusion-rate period. The three periods have been found similar in radiation and in air drying, as may be observed in Figure 7 and as was found consistently to be the case in other experiments. The conclusion, therefore, may be reached that in so far as mechanism is concerned the train of events by which a thoroughly wetted sample becomes a completely dried one is closely similar in radiation and in air drying. This same sequence has been encountered and reported in the literature for the air drying of numerous other porous non-textile materials.

Although the several drying mechanisms are distinctly different in nature, sharp discontinuities in rate curves, to separate the periods, are not always encountered. This appears to be so because, owing to water concentration gradients that become established, all parts of a material may not transfer from one mechanism to another at the same time. A gradual transition from period to period as evidenced by changes in slope of rate-regain curves will thus occur. Furthermore, nature and density of structure may be expected to affect the course of the second and third periods particularly. In this connection, Figure 9 illustrates differences in the radiation drying curves between two materials, woven wool and matted wool felt. The second, or capillary migration period is, in these instances, most affected by the structure of the samples. All experiments with these materials followed the same patterns. Densities of the two materials were about the same; the most apparent difference was a low degree of fiber alignment in the matted felt and a higher degree in the woven felt.

Each of the three drying periods deserves further discussion.

Constant Drying Rate Period

As illustrated in Figure 8, individual fibers in the constant drying rate period are completely immersed in water and an aqueous surface is presented to the atmosphere. The problem of drying here is entirely analogous to water evaporation from free bodies of water, to evaporation in humidification equipment, and the like; the water surface, rather than what lies

beneath it, is important. Water is prevented from evaporating at enormous rates by the resistance of a stagnant air film adjacent to the surface. The rate of diffusion of water through this film is dependent upon its effective thickness, which in turn is affected by the velocity, or Reynolds number, of the air sweeping past the surface. Rate of drying in this first period is constant as long as excess free water is readily available at the surface and the rate of heat supply remains constant.

The rate of drying in the constant-rate period in radiation and air drying may be satisfactorily described through normal steady-state heat balance equations. In the following radiation thermal balance, each term represents heat flow in heat units per unit area per unit time:

$$I + h(t_a - t_s) + k\Lambda(p_a - p_s) = 0. \quad (1)$$

I represents the net radiant energy input to the surface and may be expressed in terms of the surface reflectance and the lamp output, I_o, as follows:

$$I = I_0(1 - I_R/I_0 - I'/I_0). \quad (2)$$

The second term of equation (1) represents the sensible heat that flows to or from the sample because it is colder or hotter than the air; h is a gas-film coefficient; t_a and t_s are the air and surface temperatures, respectively. The final term is the heat flow in terms of the mass of water vapor that in drying diffuses through the air film from the surface, and its latent heat; k is a mass transfer coefficient; p_a is the partial pressure of water vapor in the air stream; p_s is the vapor pressure of water at the surface temperature or identically the partial pressure of water vapor in the air at the sample surface; and Λ is the latent heat. The rate of drying is related to the last term in equation (1) as follows:

$$r = -k(p_a - p_s). \quad (3)$$

Additional relations required to solve equation (1) are:

$$p_s = be^{-\lambda/RT_s}, \quad (4)$$

which is the Clausius-Clapeyron vapor pressure relationship for water, the values for which are conveniently available in the usual vapor pressure tables,

and $$\frac{h}{k\Lambda} = 0.52, \quad (5)$$

when temperature is expressed in degrees Centigrade and partial pressure in millimeters of mercury.

The normal form of the equation for gas-film heat transfer coefficient for flow parallel to a plane is:

$$h = \frac{m}{L}(Re)^n; \quad \left(Re = \frac{LV_\infty\rho_\infty}{\mu}\right), \quad (6)$$

in which m and n are experimentally determined constants.

When the radiant-heat absorption rate and the air temperature and humidity are known, then equations (1) through (6) establish the constant drying rate and the surface temperature for any velocity of air in the range for which the constants m and n are established in equation (6).

In convection air drying, radiation may, as a first approximation, be neglected and in equation (1), $I = 0$. The resulting expression is the well-known heat balance of psychrometry:

$$h(t_a - t_s) + k\Lambda(p_a - p_s) = 0. \quad (7)$$

In this instance, the equilibrium surface temperature, t_s, is called the "wet-bulb" temperature. In radiation evaporation or drying, as in equation (1), t_s also is an equilibrium surface temperature which should not, however, be confused with the wet-bulb temperature of air drying and the numerous charts, tables, and curves that are presented in the literature for the latter.

A final simplification of equation (1) is:

$$I + h(t_a - t_s) = 0. \quad (8)$$

This last equation expresses the thermal balance after drying has been completed and the material is heated to a final equilibrium temperature, t_s. The balance is between radiant-heat input and air-convection heat loss.

One method of examining the consistency of the heat balances is through the experimental determination of the constants in equation (6), relating the convection heat-transfer coefficient with air velocity. Based upon heat-transfer measurements with smooth, heated dry plates in which convective heat transfer only was important and mass transfer or drying was absent, Colburn [4] developed a correlation for the data. In the turbulent region ($Re > 20{,}000$), the following equation resulted:

$$h = \frac{0.0011}{L} Re^{0.8}. \quad (9)$$

In a definitive work on the constant drying-rate period in the drying of sand by means of convective air drying, Shepherd, Hadlock, and Brewer [17],

by using equations (3), (4), (5), and (7), and with experimental data of drying rates, sample temperature, and air conditions, determined the heat-transfer coefficient equation as follows:

$$h = \frac{0.0043}{L} Re^{0.75}. \quad (10)$$

In the present work in the constant-rate radiation drying of absorbent cotton at a number of air velocities, values of h were determined in equation (1) through measurement of the surface temperature, drying rate, and air conditions. With these data and equations (3), (4), and (5), the film coefficient for this case was established as the following function of Reynolds number:

$$h = \frac{0.0018}{L} Re^{0.8}. \quad (11)$$

A comparison is interesting of the film heat-transfer coefficient given in equations (9), (10), and (11) for convective heat transfer to smooth plates, transfer to sand dried in heated air, and transfer to a textile material dried by radiant energy with cool air sweeping the sample. The exponential coefficient on the Reynolds number is closely similar in all experiments. The proportionality constant for sand is greater than that for smooth plates, an effect which may be attributed to greater surface roughness in the former case. On this basis, absorbent cotton was intermediate in surface character, which is reasonable, provided other conditions were similar.

The procedure described above of establishing convection heat-transfer coefficients from radiation experiments and of showing an identity in the function of velocity with the coefficients determined by other means provides strong support for equation (1) as the equation for the constant drying rate period in radiation drying.

Capillary Migration Period

Between the period of drying by the evaporation of excess water, as described above, and the condition reached when the fibers contain only the saturation equilibrium content of water, the important mechanism appears to be the migration of water toward the surface by means of capillary or surface tension forces. This intermediate period is illustrated in Figures 7, 8, and 9. The general nature of capillary migration has been described by Ceaglske and Hougen [3] in relation to the drying of sand. As drying proceeds from the surface, the first action is the entry of air into the structure to replace water that has been evaporated. As air displaces water, a film of water is retained by the fibers and the familiar wick action takes place. The water moves under a force gradient which is developed by surface tension acting at the air-water interface at the junctions of adjoining fibers. If this process is very rapid, the supply of water to the surface is large enough to sustain a constant rate of vaporization. If, because of structure and surface conditions, the rate of capillary transport is relatively slow, then the capillary migration period of drying will be a falling-rate period starting at a critical regain. By way of illustration (Figure 9), matted wool felt has an almost constant drying rate during the capillary migration period as the saturation equilibrium regain (about 0.54) is approached. In the woven wool felt, on the other hand, a more commonly found decline in rate during the migration period is apparent, starting at a distinct critical regain of about 2.0. In Figure 7, it is evident that this second or intermediate period of drying is present in both radiation and air drying. The rate-regain curve for the period is not now capable of prediction for a new material; it must be determined experimentally.

Direct experimental evidence for capillary migration was gathered from the following simple experiments. Pieces of 0.66-centimeter white matted wool felt, about 8 centimeters square, were thoroughly wetted in boiling water and were cooled to room temperature. They were then squeeze-rolled to a regain of 1.5, which is well above the equilibrium saturation value, excess free water remaining on the fibers. By means of a fine hypodermic needle, a small quantity of ink was deposited through the edges of the felt into the central interior of its structure. A number of such deposits were made in several squares of felt. One sample of felt so treated was dried in the radiation drier and another in an air-drying oven at 110°C. Still another sample was placed between sheets of waxed paper to retard surface evaporation. This sample served as a blank. After 12 hours, cross-section explorations were made with a razor and the migration characteristics of the ink were studied. Typical effects are shown diagrammatically in Figure 10. In the blank sample, which was protected from surface drying, the ink spot spread uniformly in a typical diffusion pattern. In samples dried by either infrared or air a very dense ink film was found to be deposited at the

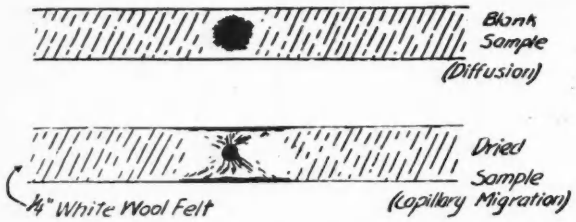

FIG. 10. *Comparative effects of capillary migration and diffusion.*

surfaces. This is the result to be expected from capillary migration of ink and water to the surface during drying which leads to a concentration and deposition of the ink pigment at the surface. It is interesting to note that capillary migration is part of the drying procedure regardless of the means of applying the necessary heat energy.

Because capillary migration is an hydraulic flow, it becomes an obvious inference that any material dissolved or suspended in the water, such as a salt, a dye, or a resin finishing agent, will also be transported to the outer surface and will be concentrated there by the evaporation of water. In the extreme case, undesirable effects such as harsh hand, non-uniform dyeing, or surface hardening may result, with the extent of the effect roughly proportional to the thickness. To minimize surface concentrations of impurities when these are objectionable, the possibility exists for circumventing the capillary diffusion drying period by placing the textile in question between the equivalent of blotting or leader cloths (Figure 11). Excess free water and its content of dissolved or suspended material flows into the receiving leader cloths and thus is effectively removed prior to a subsequent drying operation. Preliminary studies with blanket material moistened with 5-percent sodium chloride solution indicated that a relatively softer product resulted when blotting preceded drying than when it did not. For a similar procedure in the field of leather drying, reference is made to U. S. Patent 2,131,645 issued to T. K. Sherwood and L. E. Garono.

Diffusion Period

The final period of drying takes place after the regain has been reduced to less than the saturation equilibrium value, about 0.54 for wool. The rapidity with which the rate of drying decreases during this period, in the face of rapidly rising temperatures (see Figures 7 and 8), is evidence that the processes for

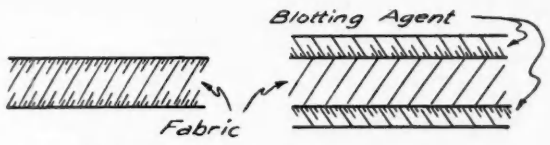

FIG. 11. *Method of removal of water and dissolved or suspended material by capillary flow.*

removing the final moisture are difficult compared to the preceding drying periods. The characteristic of a rapidly falling rate with decreasing moisture content applies equally to radiation and air drying. This final period of drying is the period of greatest practical interest.

Diffusion-rate drying may be viewed as a sequence of events in series by which heat is introduced through the fabric structure to any given fiber and the vaporized water, released from the fiber with an absorption of heat, diffuses to the surface of the fabric. Both the bulk structure of the fabric with its air pockets and the moisture characteristics of the fiber are involved in the processes.

In air drying the heat transfer proceeds by means of conduction through the gas film surrounding the fabric and then through the complex path of fibers and air pockets within the structure. In radiation drying, energy is absorbed at different distances from the surfaces. Once converted to sensible heat, the radiation energy is, of course, transferred by conduction as in air drying. The interesting possibility exists that, because of transmission of radiant energy to some depth into a fabric, a more favorable temperature and moisture gradient for drying might result.

Two processes are involved in the mass transfer of water vapor—that is, diffusion from within a fiber to its surface and diffusion through air from fiber surface to outer textile boundary. Present experiments were not designed to impart fundamental information regarding these processes. However, moisture migration in textile fibers has been discussed in the literature. For example, King and Cassie [12], through water-sorption experiments with wool in a vacuum, eliminated the effect of air diffusion. They found the rate of moisture adsorption to be extremely rapid and to be limited primarily by the rate at which the heat of vaporization could be dissipated in the vacuum. The time required for the fibers to come to equilibrium, after allowing for temperature changes, was estimated by the authors to be less than 15 seconds. Presumably, desorption of moisture in

a vacuum would be equally rapid. Because the rate of drying of a bulk textile in the presence of air is very slow compared to the fiber rate processes, diffusion *within* the fiber probably is not important and the controlling rate most likely is the diffusion of water vapor through air to the fabric surface. Although mathematical analyses for diffusion from slab-like materials are available [9, 20] and studies of water-vapor permeability of textile materials have been performed [2], quantitative analysis of the diffusion problem is rendered difficult because temperatures as well as composition are changing rapidly. Generally, therefore, the effect of variables which are diffusion rate-dependent, such as sample thickness, is determined empirically.

Drying Temperatures and Energy Relationships

It is interesting to compare temperature histories in typical radiation and air-drying experiments. These are supplied in Figures 7 and 9. In the first drying-rate period, temperatures are approximately constant and are established by the heat balances of equations (1) and (6). Although rates of drying may remain constant or fall appreciably in the second or capillary migration period, the temperature continues practically constant. This indicates no difficulties in supplying latent heat of vaporization in the period, regardless of textile structure, although there are differences in the free availability of water for vaporization. Concurrently with a rapid decrease in drying rate, as the third drying period is entered, a rapid rise in temperature sets in. After drying has been completed, an air-dried sample achieves the temperature of the air, and a radiation-dried sample achieves a final temperature that is dependent upon the film heat-transfer coefficient and rate of radiant heat input, as shown in equation (8). The final radiation temperatures may be high and can easily exceed fiber decomposition levels. It must also be noted that because of large energy inputs in radiation drying the time rate of change of temperature when moisture content is low is much greater than in air drying. There is apparent need for rapid-acting and adequate controls to limit the drying operation, and to avoid excessive temperatures at the end of the operation in radiation drying.

Figures 12 and 13 are presented to permit comparison between distribution of energy consumption in radiation and air drying. The figures were computed from experimental data but are to be taken only for purposes of qualitative comparison. One hundred percent heat in Figure 12 for radiation drying includes all radiant heat adsorbed by the sample and does not include energy lost by reflection or transmission through the sample. Until the temperature begins to rise at a regain of 0.8, approximately 87 percent of absorbed energy is used for vaporization, the remainder being lost to the atmosphere. This convection loss existed because the sample was subjected to a stream of room-temperature air. When heated air is used in conjunction with radiation as in practice, a vaporization efficiency greater than 100 percent based only on radiant input as defined for Figure 12 is obtained. As regain decreases below 0.8, energy used in vaporization decreases, and an increasing amount is used as sensible heat to raise the temperature of the sample. Losses to the atmosphere by convection increase markedly until, at dryness, only convection loss remains.

In air drying, illustrated in Figure 13, the heat of vaporization is the sole heat demand upon the convection-supplied energy until a regain of 0.8 is reached. At lesser regains, temperature rises, as in radiation drying, and part of the heat is employed as sensible heat, the proportion of sensible heat to energy input increasing continually, until, at the final

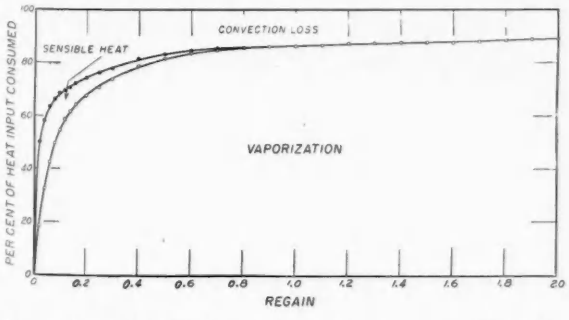

Fig. 12. *Distribution of energy consumption in radiation drying.*

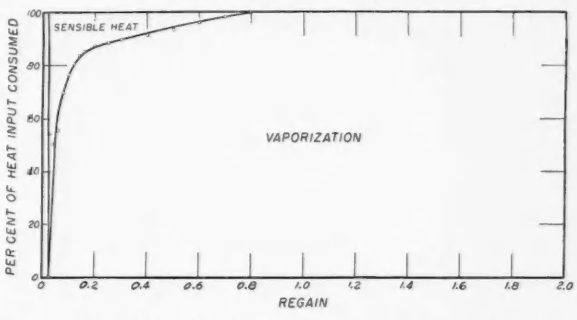

Fig. 13. *Distribution of energy consumption in air drying.*

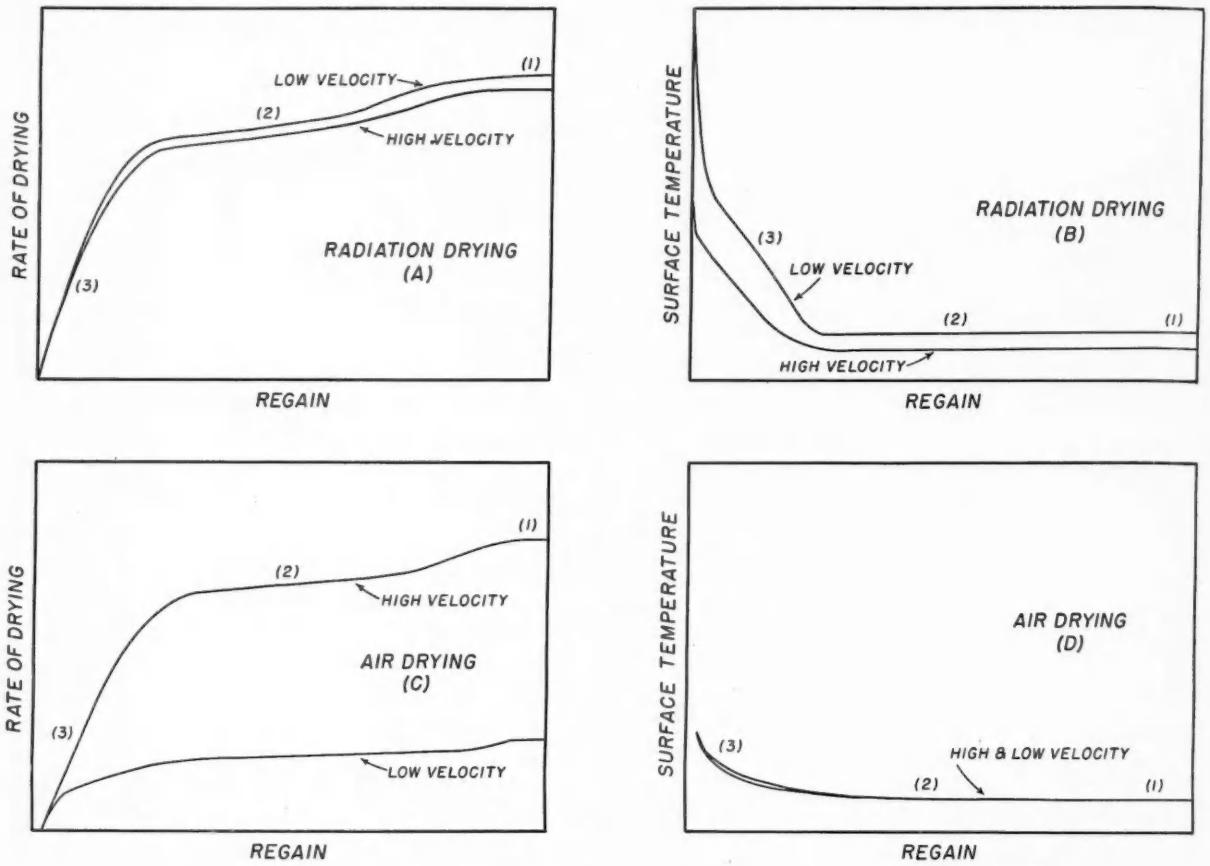

FIG. 14. *Relative effect of air velocity variation on radiation and air drying.*

equilibrium regain and temperature, both become zero.

Effect of Individual Variables

This section deals with experimental results and is intended to show, as far as possible, the effect of individual variables in radiation and air drying. Experiments, mainly with felted materials, were arranged to yield information, within the limits afforded by the drying equipment, regarding the effect of major variables upon the course of drying. Variables included air conditions, intensity of radiant energy, sample thickness, optical properties such as absorptivity, reflectivity, and transmissivity, structure of the textile material, and, finally, the effect of initial regain upon drying rates.

Air Conditions

Air Velocity. A composite view of the relative effect of air velocity on rate of drying and on sample temperature in radiation and air drying is presented in Figure 14. The curves were obtained by interpolation and extrapolation of drying data for matted wool felt and show, for a comparable set of conditions, the consequence of high and low air velocities, 4.0 and 0.8 meters per second, respectively, upon rate of drying and surface temperature. The comparable conditions were equal rates of drying and equal surface temperatures in the first period and at a velocity of 4.0 meters per second.

It is observed in Figure 14A that in radiation drying a substantial change in air velocity has a minor effect upon the rate of drying. Also, as expected, the rate of drying in the third period, being an internal process, is even less affected by external air parallel to the sample surface. In the initial constant-rate period the relation between rates at different velocities is expressed by equation (1). Although convection effects are present through variations of h and k with velocity, the radiant heat input, I, overshadows these, and the net effect of velocity is small. The fact that the rate of drying at high air

velocity is lower than that at low air velocity results from the experimental condition that room-temperature air was used in the present experiments. As air at a higher temperature is employed, the relative position of the two curves will reverse, but the effect of velocity will become important in periods 1 and 2 only when very hot air is used in conjunction with a radiant heat supply. Three sets of rate data showing the relatively small effect of air velocity and a generally decreased drying rate with increased air velocity are given in Figures 15, 16, and 17 for matted wool felt, woven wool felt, and absorbent cotton.

A series of drying runs was performed with woven wool felt solely in the constant-rate period with different air velocities. Through the use of especially high initial regains, extended constant-rate periods with improved precision were obtained. These drying rates versus air velocity are plotted in Figure 18. The curve in this figure was computed from equation (1) and the agreement is considered satisfactory. The rate value at zero duct velocity indicates that because of convection the actual air velocity was substantial. In computing the curve, the constants, I, h, and k, were evaluated through measured values of drying rate, air temperature, partial pressure, and surface temperature at a velocity of 1.3 meters per second and through the use of equations (1), (3), (4), and (5). The remainder of the curve was then computed through the additional use of equation (6).

Figure 14B illustrates typical behavior of the sample surface temperature in radiation drying at low and high air velocities. The temperature remains substantially constant during the first two periods, with an actual slight decrease in rate because of change in surface reflectivity as regain decreases. A rapid rise in temperature ensues in the third period with termination at dry-sample temperatures defined by equation (8). Figure 19 presents temperature-regain data in the drying of matted wool felt at different air velocities. Surface temperatures only are presented here. The fact of decreasing surface temperatures with increasing air velocities is the result of using cool (room temperature) air in the experiments. When the air is hotter than the surface temperature, then velocity will have a reverse effect. The effect of velocity on surface temperature may be estimated in period 1 through equation (1). In Figure 20, points represent measured surface temperatures in the drying of absorbent cotton, with the solid line computed by means of the equation.

As is well known, air velocity is a major variable in the drying of materials by means of convection air. Figure 14C illustrates the large difference in drying rates between the high and low air velocities. The shapes of the curves and the divisibility into three zones are the same as in radiation drying (*cf.* Figure 7). The constant drying rate period may be described through equation (7). This has been discussed amply in the literature [17].

Temperature-regain characteristics during air drying are illustrated in Figure 14D. Wet-bulb temperature is maintained substantially constant and independent of air velocity during the first period, with a slight rise in the second period. A rapid increase in temperature during the diffusion period ends finally when the sample achieves the air temperature.

Air Humidity and Temperature. No experiments were performed in which the temperature and humidity of the air stream were varied during radiation drying. Since all complete drying curves are "anchored" in the constant-rate period, it is instructive to compute, through equation (1), the effect of humidity (or partial pressure) and air temperature in this period. The results of such computations are given in Tables I and II.

Table I shows that, for the set of initial conditions listed, the effect of water vapor in the air, when the latter is essentially at room temperature, is minor both with respect to surface temperature and to rate

TABLE I. COMPUTED EFFECT OF VARYING AIR HUMIDITY
t_a = 25.6° C; I = 1,010 g.-cal./m.² × sec.; Λ = 590 g.-cal./g.; air velocity = 1.3 m./sec.

Partial pressure of water vapor in air (mm. Hg)	Surface temperature (°C)	Rate of drying (g./m.² × sec.)
0	52.5	1.48
12	54.5	1.47
24 (sat.)	56.3	1.46

TABLE II. COMPUTED EFFECT OF VARYING AIR TEMPERATURE
p_a = 12 mm. Hg; I = 1,010 g.-cal./m.² × sec.; Λ = 590 g.-cal./g.; air velocity = 1.3 m./sec.

Air temperature (°C)	Surface temperature (°C)	Rate of drying (g./m.² × sec.)
25.6	54.4	1.48
46	56.0	1.61
54	56.7	1.68
71	57.7	1.80
93	58.3	1.96

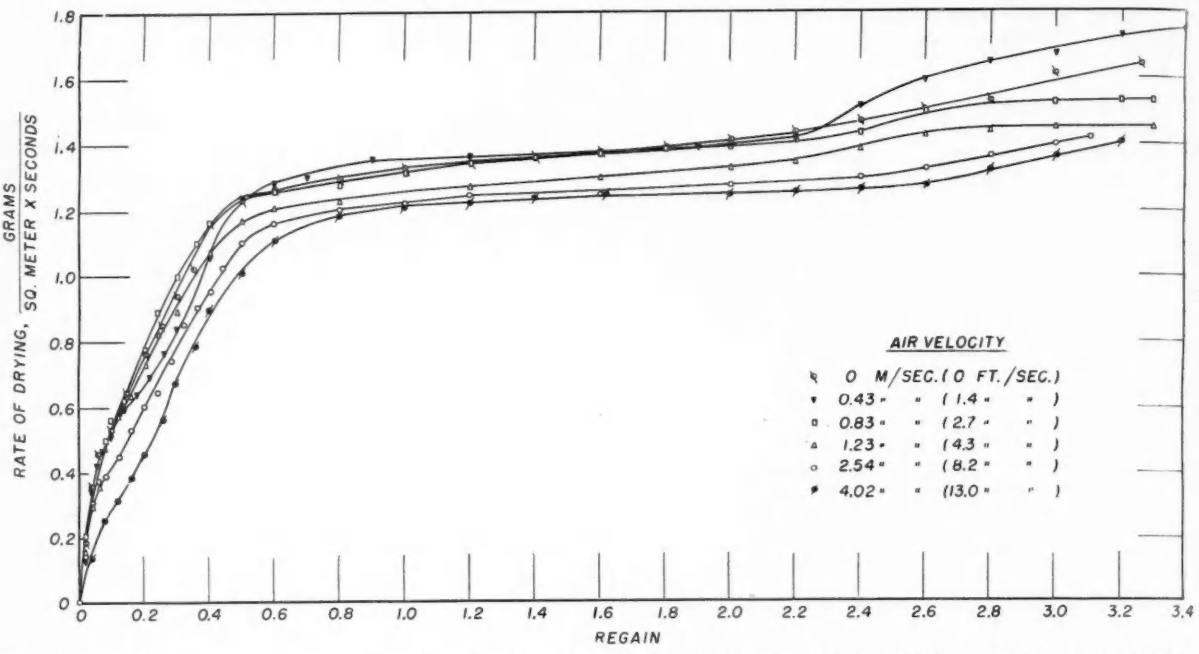

FIG. 15. *Rate of drying versus regain. Effect of cool air velocity in radiation drying of matted wool felt.*

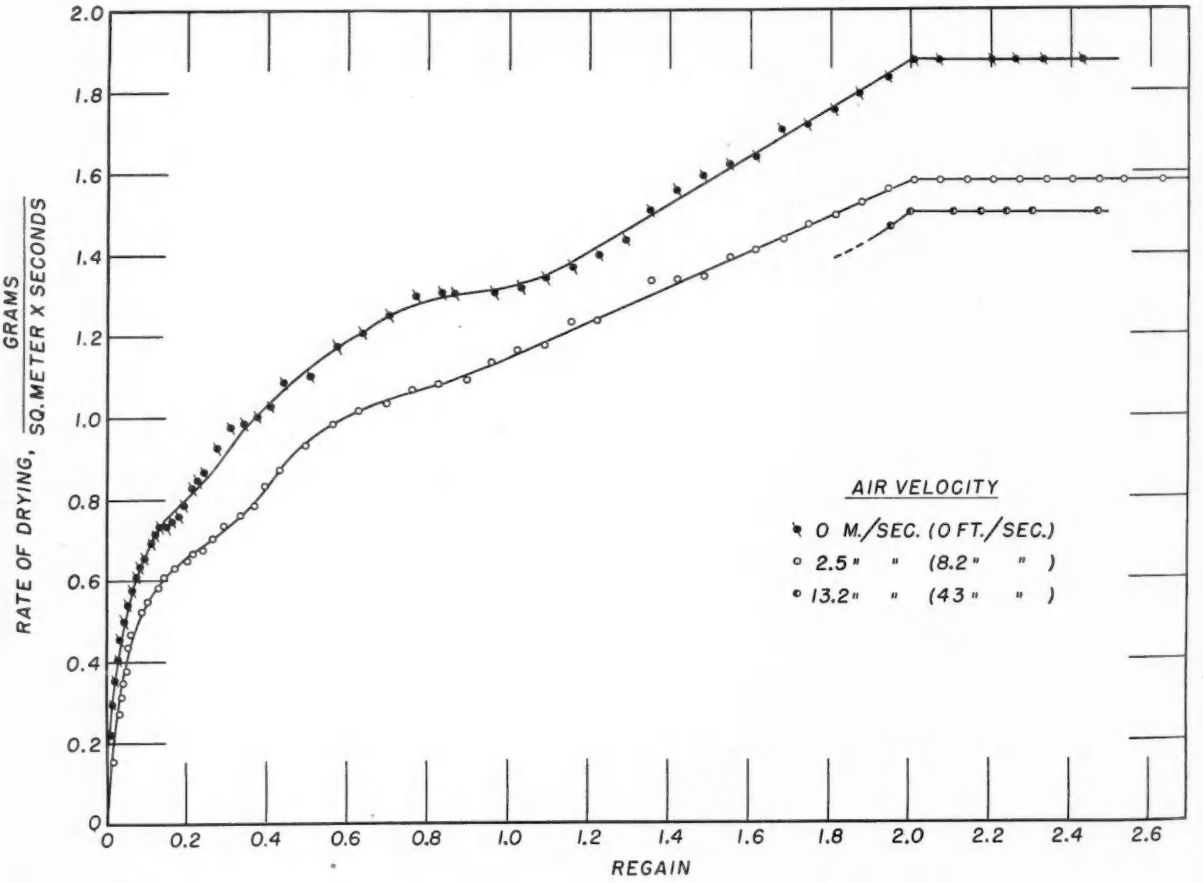

FIG. 16. *Rate of drying versus regain. Effect of cool air velocity in radiation drying of woven wool felt.*

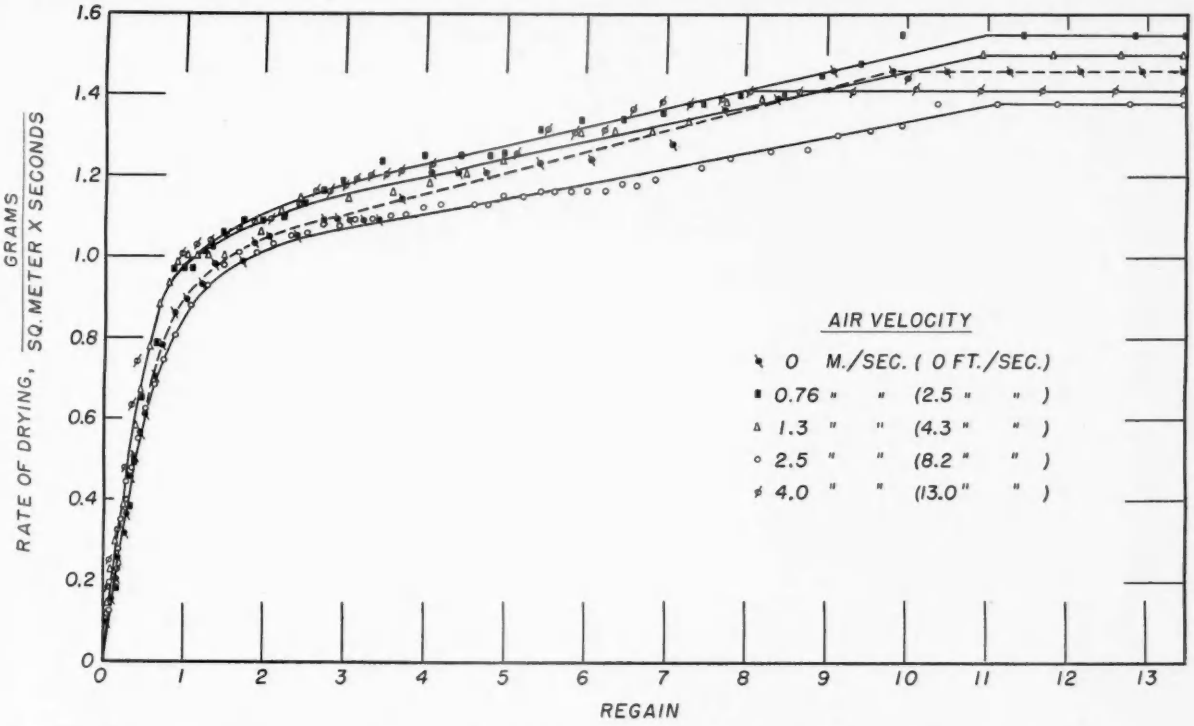

FIG. 17. *Rate of drying versus regain. Effect of cool air velocity in radiation drying of absorbent cotton.*

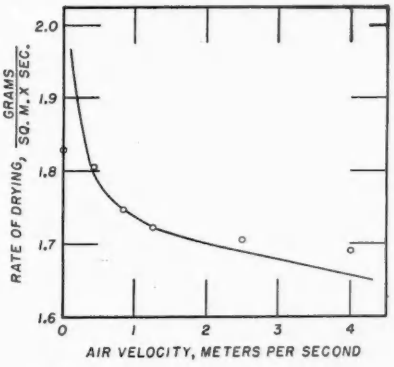

FIG. 18. *Effect of cool air velocity in constant drying-rate period of radiation drying of woven wool felt. Line is computed and points are experimental.*

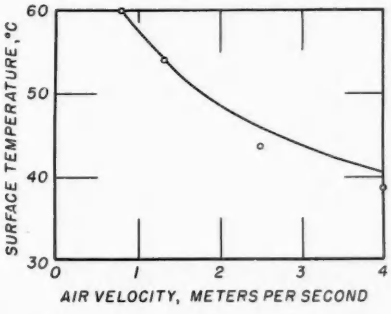

FIG. 20. *Effect of cool air velocity in constant drying-rate period of radiation drying of absorbent cotton. Line is computed and points are experimental.*

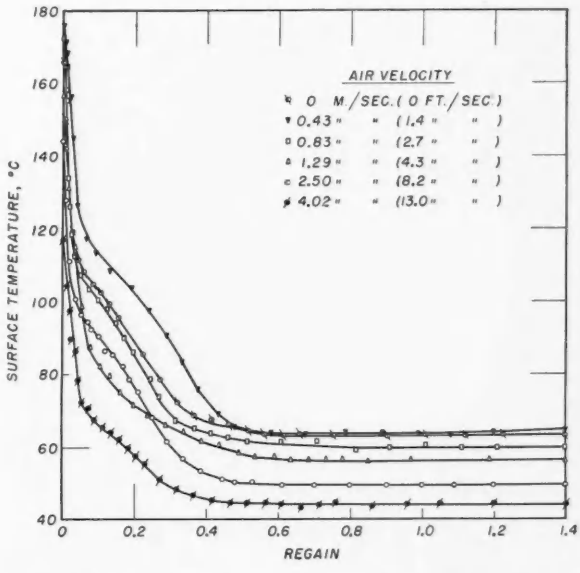

FIG. 19. *Temperature versus regain. Effect of cool air velocity on surface temperature in radiation drying of matted wool felt.*

of vaporization. This results from the fact that the water content of air from zero humidity to saturation at room temperature is a small number compared to the equilibrium partial pressure on the surface of the sample (*cf.* equation (3)).

Table II shows that surface temperature does not change appreciably for a rather wide range of selected air temperatures. The rate of evaporation does increase somewhat, a 21-percent rise resulting from an increase in air temperature from 25.6° to 71°C. As the temperature is increased, a point is reached (56.9°C under the given conditions) at which surface and air temperatures are equal. Here all the radiant energy absorbed is used in the vaporization of water and none is lost as sensible heat. Below this temperature some of the radiant flux is lost in the form of sensible heat and above it the radiant energy entering the sample is supplemented by sensible heat from the gas stream. Hence, above this temperature there exists the combination of a radiation drier and a normal convection drier.

The effect of humidity and temperature in the first drying period in air drying is well known, is determined through equation (7), and needs no amplification.

Intensity of Radiation

Intensity of radiation striking the sample was controlled by variation of the spacing between lamp banks. Drying tests were run at six incident intensities thus obtained. A logarithmic plot of intensity, calculated from measured pyrheliometer readings, against the distance between the banks is presented as Figure 21. The relation is shown by a straight line with a slope of -0.62. (For a point source of radiation the slope would be -2, *i.e.*, the inverse square law; for an infinite lamp bank, the slope would be 0.)

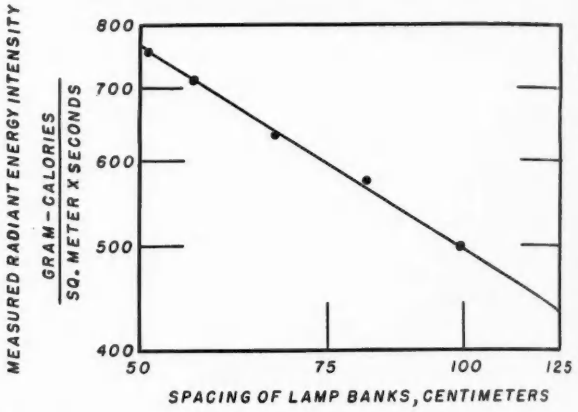

FIG. 21. *Relation between spacing of lamp banks and measured intensity of radiation.*

Drying rates versus regain relations at different intensities are presented in Figure 22. It is apparent that within the range of incident intensity covered (440 to 760 gram-calories per square meter second) the curves are similar in shape at regains above 0.6, the approximate regain at which the controlling mechanism changes from capillary migration to diffusion. In the diffusion-controlled period, although

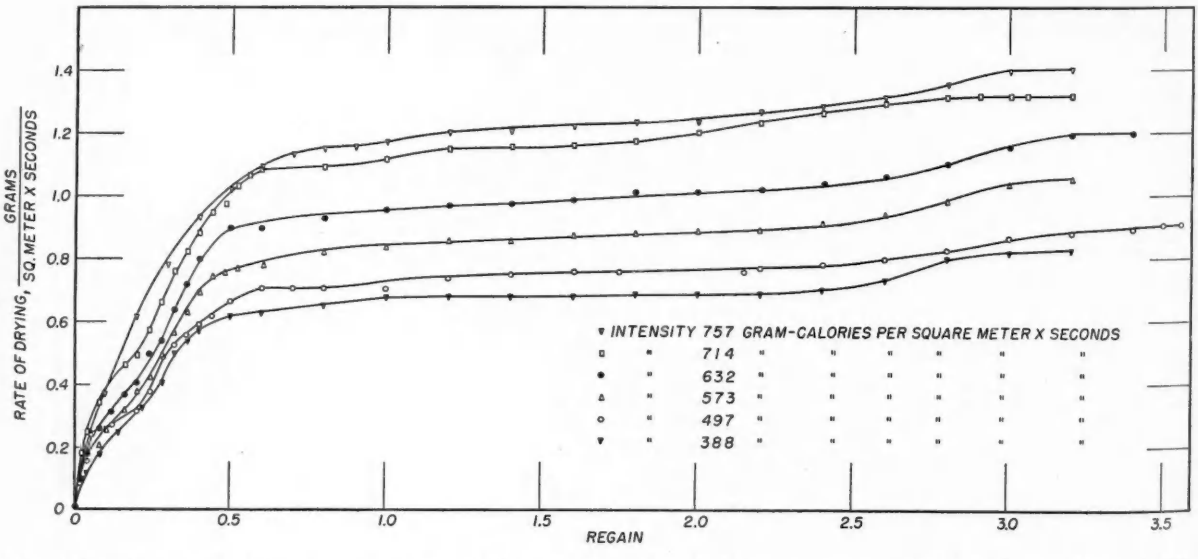

FIG. 22. *Rate of drying versus regain. Effect of intensity of radiant energy on drying of matted wool felt.*

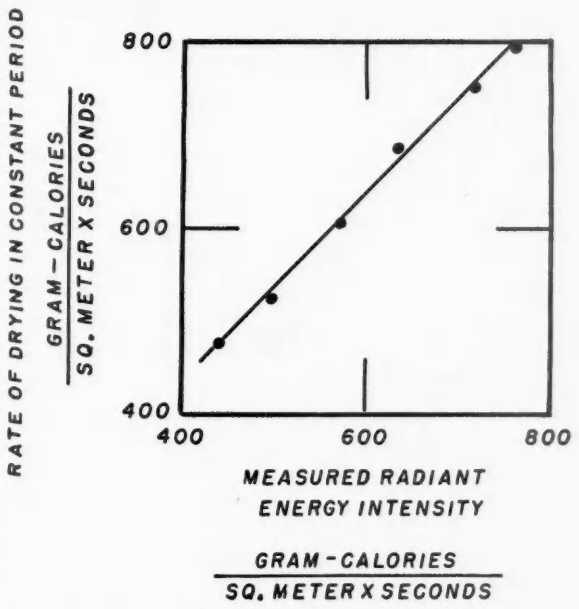

FIG. 23. *Rate of drying (energy equivalent) versus measured radiant energy intensity in the constant-rate period of radiation drying of matted wool felt.*

the drying curves do not all remain clearly separated, it is apparent that drying rates remain essentially proportional to intensity.

Drying rates in the initial substantially constant-rate period, calculated in terms of gram-calories per square meter second, are plotted in Figure 23 against the measured incident intensities in the same units. Rates are proportional to intensities, but it is indicated that energy output in evaporation alone, exclusive of convection loss, consistently exceeds apparent input by about 5 percent. Allowing for the convection loss of about 12 percent of the absorbed energy, excess total energy output over measured incident intensity is approximately 20 percent.

An explanation for this discrepancy is necessary. Several possibilities were investigated: experimental errors in measurement of either sample weight or radiation intensity; incorrect calibration of pyrheliometer used to measure radiation intensity. The consistency of the discrepancy makes unlikely experimental errors in sample weight which would not be constant. Radiation intensity distribution was studied, and the maximum variation over the area of the sample was found to be 6 percent, which is far too small a variation to explain the discrepancy. This discordancy points to a constant error, for which reason the pyrheliometer calibration was investigated.

Calibrations of both pyrheliometer and thermopile are not satisfactory because of the complete absence of reference standards in the intensity range employed. Standards used in calibrating the thermopile exposed it to intensities between 0.4 and 19×10^{-6} watts per square millimeter. A straight-line relation between intensity and potential difference existed in this range, and was therefore assumed to be valid to the radiation intensity of 2×10^{-3} watts per square millimeter employed in this study (or one hundred times the maximum intensity employed in calibration), a very uncertain extrapolation.

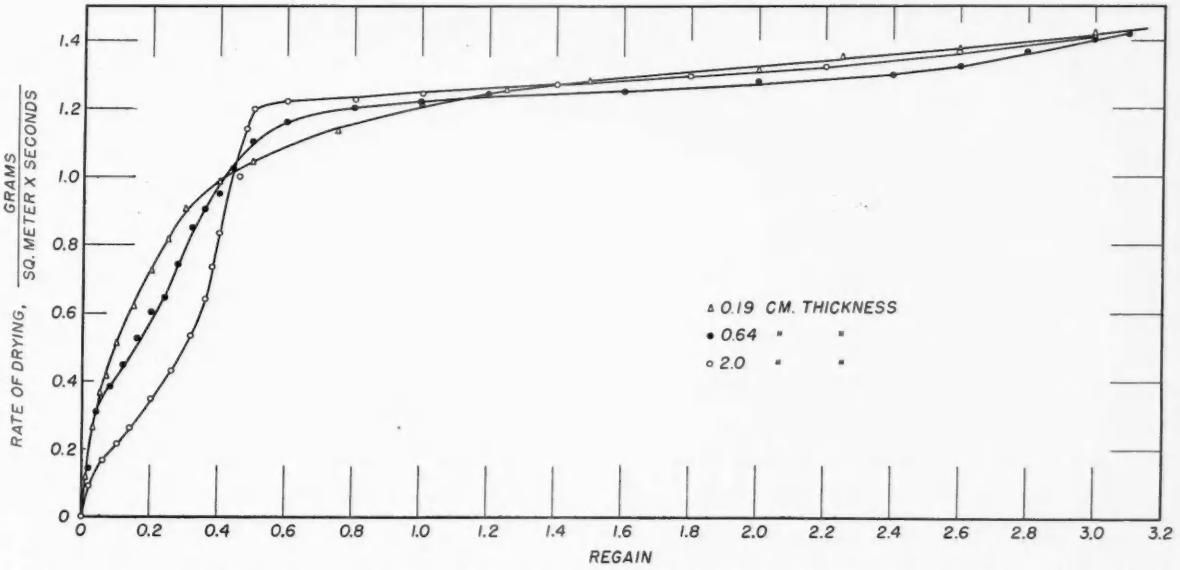

FIG. 24. *Rate of drying versus regain. Effect of sample thickness in radiation drying of matted wool felt.*

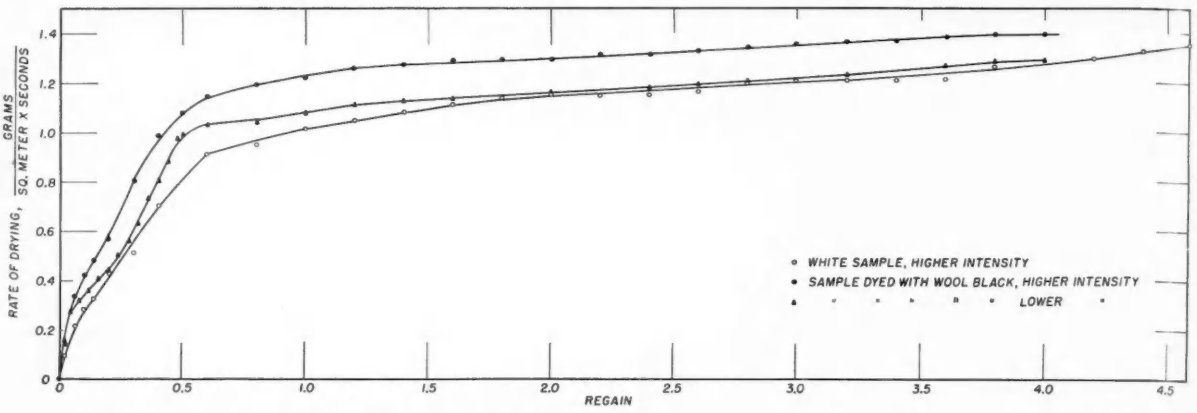

FIG. 25. *Rate of drying versus regain. Effect of sample color in radiation drying of matted wool felt.*

The situation with respect to the pyrheliometer was even less satisfactory. According to the description of this instrument in the literature [11], "voltage is very nearly proportional to the intensity of the solar radiation." Its calibration by its manufacturer was based on the assumption that this relation was strictly proportional, but no individual data were available to make a statistical analysis of the calibration with the purpose of determining reliability of the extrapolation. In using this instrument, intensities were encountered which were ten times that of sunlight, or the maximum employed in calibration, and the strict proportion assumption was perforce used. This procedure is capable of producing errors of the magnitude of those observed.

Need for higher-intensity primary standards for calibration has been expressed by the manufacturer * in a statement with which the authors concur. Certainly their availability would have improved the reliability of the intensity portion of this study.

Sample Thickness

Rate-regain curves for three thicknesses of matted wool felt dried by means of radiation are shown in Figure 24. The sample thicknesses were 0.25, 0.60, and 1.9 centimeters.

The rates of drying from regains of 3.0 to 1.0 have the same value for all thicknesses. This fact indicates that the second, or capillary transport period, which predominates in this regain interval, is one in which surface evaporation is most important. The sharpness of the critical value of the moisture content between capillary transport and the diffusion period varies considerably with sample thickness. The thinnest sample decreases in rate gradually and the thickest sample, abruptly. Within the final period, however, the thinnest sample is favored by the highest rate.

The general form of the rate curves in the diffusion period is consistent with previously recorded [14] findings for the air drying of paper pulp; namely, concave upward for thick samples and concave downward for thin.

Optical Properties

Absorptivity, Reflectivity, Transmissivity, Color. Related to the intensity of illumination striking the sample is the proportion of that illumination absorbed by the sample—that is, the absorptivity $(1-I_R/I_o)$, as described in equation (2). Absorptivity of a fabric is dependent upon its composition, fiber shape, thickness and density, color, and regain. In a single comparison, constant-rate drying rates of white absorbent cotton and white matted wool felt at equivalent drying conditions differed by less than 5 percent. It has long been known that sample color affects absorptivity—that is, that absorptivity is a function of the wave lengths of the radiation. It has recently been shown [1] that materials with similar reflectivity characteristics in the visible range of the spectrum—that is, which look alike to the eye—may differ greatly in the near infrared.

Effect of sample color in radiation drying was investigated by the use of extremes: a white matted wool felt, and the same felt dyed with Wool Black 4B, a dye with a comparatively high infrared reflectivity, and with a comparatively low infrared-reflective black dye mixture containing Calcocid

* R. Anderson, Manager, Eppley Laboratory, private communication.

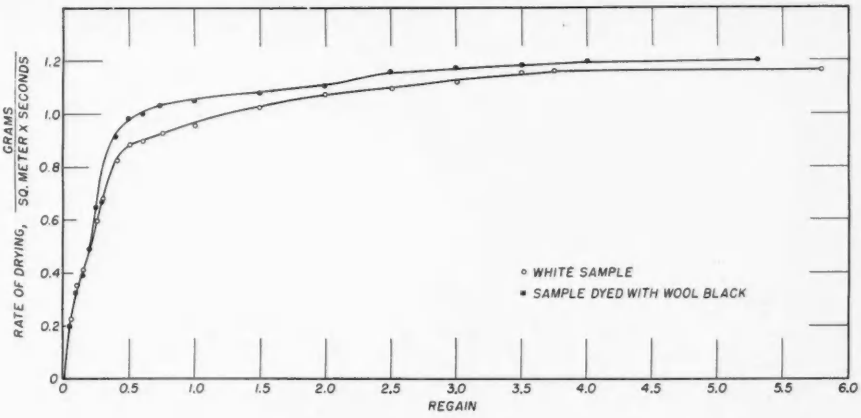

FIG. 26. *Rate of drying versus regain. Effect of sample color in radiation drying of woven wool crepe.*

Green B. Reflectivity of these samples was tested by means of a 2,500° K lamp source and a thallium sulfide cell effective in the range 7,500 Å. to 11,000 Å. As drying proceeded from dripping wetness to a regain of less than 0.03, the reflectivity of the white sample rose from 57 to 87 percent, that of the wool black-dyed sample from 27 to 42 percent, while that of the sample dyed with the Calcocid Green containing the black mixture remained between 12 and 10 percent. In the thicknesses used (0.6 to 0.8 cm.) transmissivity was in all cases less than 1 percent, and absorptivity was substantially 100 percent less the reflectivity. Independent reflectivity measurements on white absorbent cotton by means of the Eppley thermopile showed this property to increase with decreasing regain in the constant-rate period, in spite of greatly increased surface roughness as drying proceeded.

Contrasting samples of white and black-dyed felts were dried at the same conditions of intensity, air velocity, and temperature. Through most of the capillary migration period the samples dyed with wool black and Calcocid Green B dried, respectively, about 11 and 21 percent faster than the white samples and were, respectively, about 12°C and no hotter. During the diffusion-controlled period the black samples dried about 45 and 48 percent faster, respectively. At final dryness the blank samples were about 19° and 14°C hotter. Figure 25 presents the data on the white and the wool black-dyed samples.

For further comparison of white and black samples, the lamp spacing, and hence the radiation intensity, was adjusted between white and black sample runs so that the initial drying rates at high regains were identical. The result is included in Figure 25. The rates differed by less that 2 percent to a regain of 1.8, and the black sample was about 5°C cooler. Below this regain the black sample was found to dry more rapidly than the white sample.

For a test of conditions at which transmission became important, there was compared the drying behavior of equal radiation intensities of a 0.85-mm. woven wool crepe, white, and dyed with wool black. Results are given in Figure 26. They were qualitatively similar to those obtained with felts, and showed the flatter curve for the black sample in the late capillary flow period. It is apparent that the absorptivities of the differently colored materials do not follow similar courses. The conclusion is drawn that, with 2,500° K radiation sources and intensities between 400 and 800 gram-calories per square meter second, drying rates of cloths at high regains are not appreciably sensitive to dye characteristics, but are significantly sensitive at the lower regains encountered in drying practices. This effect is probably due to the absorptivity of water for infrared radiation.

In the comparison of radiation-drying and air-drying runs, it was observed that sample temperatures were not entirely constant from the initial warm-up to the end of the capillary-drying period. As drying proceeded, the air-dried samples consistently became about 2 degrees warmer, but the radiation-dried samples usually became a few degrees cooler. Figures 4, 6, and 7 illustrate this behavior. The increase in sample temperature in air drying may be the result of increasing difficulty of capillary migration to the zone of vaporation, a condition for which steady-state equation (1) would not be valid.

The usual observed decreasing surface temperature in the first two periods of radiation drying is consistent with the measured increase in reflectivity, as may be calculated from equations (1) and (2). The increased I_R results in a decreased value of I. For the equation to be satisfied, the sum of the other

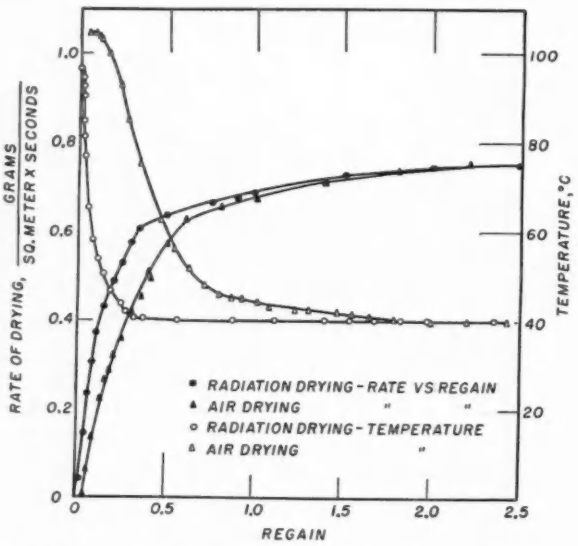
RATE OF DRYING, GRAMS / SQ. METER X SECONDS
TEMPERATURE, °C
REGAIN
RADIATION DRYING - RATE VS REGAIN
AIR DRYING " "
RADIATION DRYING - TEMPERATURE
AIR DRYING "

FIG. 27. *Rate of drying and temperature versus regain. Comparison of radiation and air drying of matted wool felt at comparable rates of energy absorption as measured by equal initial drying rates when thoroughly wet.*

two terms must decrease, and the only variables which can respond are t_s and therefore p_s. Both decrease.

There is evidence that radiant transmittance, or penetration, may be important to the internal temperature of thick textile materials during heating or curing, especially when the regain is low and when no large heat-absorbing process, such as vaporization or drying, is present. Although subject to experimental error of placement, thermocouples in the center of felt samples during radiation drying generally were found to indicate a higher temperature than those at the surface, a result expected if any appreciable optical penetration occurs. The results shown in Figures 4 and 6 are typical. In the first and second drying periods, the center temperature was only modestly higher than that of the surface (up to about 7°C between different runs). After drying was completed a considerable and more significant difference between center and surface was observed (up to 30°C between different runs).

Direct evidence for radiant penetration was obtained by heating to excess a dry, close-pressed pile of eight 0.19-cm. pieces of gray matted felt. They were exposed to high-intensity radiation on each outer surface and these surfaces were subjected to a parallel stream of air at room temperature. Using degree of decomposition as evidence of the relative temperature level attained, it was strikingly apparent that the innermost layers were most strongly singed, the outer layers appeared unaffected, and others were graduated between. Experiments were also performed to carbonize cellulose burrs in raw wool with radiant energy and here, too, it was found that particles in the center of a layer darkened and became brittle more rapidly than those on either surface. The carbonization was performed by soaking the wool in 4°Bé sulfuric acid, centrifuging, and heating between large banks in the usual manner.

With respect to the effect of radiation penetration on rate of drying, through possible redistribution of internal moisture gradients, some judgment may be made from the comparative results in Figure 27. Air-drying and radiation-drying experiments with 0.19-cm. gray matted wool felt were performed under conditions such that equal drying rates prevailed when the samples were very wet. At regains less than 1.0, the drying rates for the two methods of drying diverged and the radiation-drying rate was larger. Before this regain had been reached, the temperature of the air-dried sample had begun to rise while that of the radiation-dried sample remained constant, resulting in a higher drying rate at a lower temperature for the radiation-dried sample. This combination is consistent with the existence of some radiant penetration. Similar results were obtained in comparison dryings of 0.64-cm. matted wool felt.

Quantitative aspects of the optics of textile materials will be presented in a subsequent paper.

TABLE III. CHARACTERISTIC REGAINS OF DIFFERENT TEXTILE STRUCTURES

Material	Thickness (mm.)	Dry "weight" (kg./m.²)	Regains: Initial*	Regains: First critical†	Regains: Second critical‡
Woven wool felt	15	5.1	2.1	2.0	(0.5)
	9	3.1	2.6	2.0	(0.4)
Matted wool felt	19	4.8	3.5	(3.1)	(0.5)
	6.4	2.1	3.5	(3.0)	(0.4–0.6)
	2.5	0.43	6.6	(6.0)	(0.4–0.6)
Woven wool crepe	0.85	0.18	6.1	3.8	(0.5)
Absorbent cotton	5 (wet)	0.53	16.5	10.7	(1–1.5)

* Sample horizontal, "dripping" wet.
† Between periods 1 and 2.
‡ Between periods 2 and 3.
All values in parentheses are approximate.

Materials

The nature of the textile material affects the course of the drying operation. As has been illustrated in Figures 9, 17, and 26, drying-rate curves of matted felt, woven felt, absorbent cotton, and woven crepe show broadly similar characteristics indicating similar drying mechanisms. The curves differ, however, in value of initial regain (at dripping wetness), prominence of constant-rate period, distinctness of critical regains, and duration of capillary falling-rate periods.

The materials possessed a variety of bulk structure properties of importance in drying. The effect of these different structures on characteristic regains is shown in Table III.

All values are averages and are independent of the method of supply of energy—that is, whether air drying or radiation drying is employed.

Among the wool samples, initial regains and first critical regains are dependent upon texture, whereas the second critical regain is independent of it, being a result of the saturation equilibrium regain of the material at the approximate temperature and 100 percent relative humidity. All thicknesses of the matted wool felt were found to have very indistinct and brief initial constant-rate periods. These felts displayed, moreover, one, or frequently two, quite distinct periods of very nearly constant drying rate within the capillary migration period. The woven crepe has a similarly high saturation regain but, in contrast, a long constant-rate period. It therefore possesses either a relatively thick initial water surface or a bulk structure permitting ready flow of liquid, more probably the latter.

Absorbent cotton has an extremely loose structure and consequently an enormous water capacity. Its constant-rate period is distinct and long, owing to a continued ready supply of water by capillary flow. By visual observation, its thickness remained constant until the sample reached a regain of about 0.5, well within the diffusion-controlled period, after which its structure became expanded. In the diffusion-controlled period, graphs for different air velocities converge very closely.

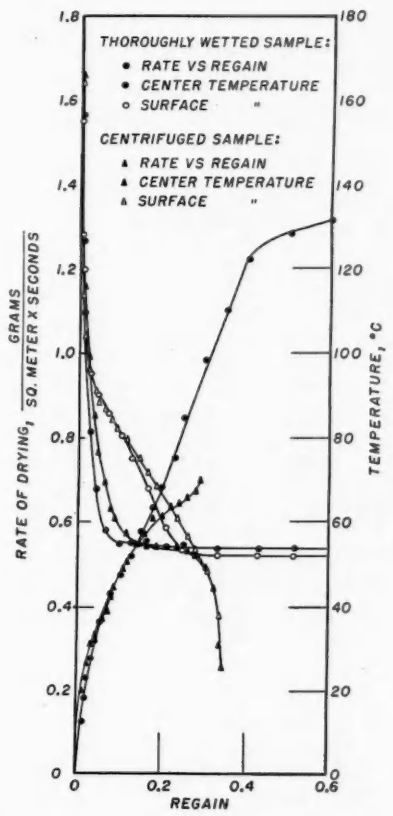

FIG. 28. *Rate of drying and temperature versus regain. Effect of initial regain upon the course of radiation drying of matted wool felt.*

Initial Regain

Most experiments in this work started with a fully wet sample for theoretical reasons. In industrial practice as much water as possible is removed by centrifuging or pressing prior to drying. As a result, the stock is usually in the falling-rate period at the beginning of the drying process. To obtain this condition, therefore, felts were centrifuged to between 0.25 and 0.40 regain and then dried, four by means of radiant energy and two by means of heated air. In the region of common regain these samples, once heated, had drying rates that did not differ greatly from rates obtained with samples thoroughly wetted initially. However, the shapes of the curves were different, doubtless because of different moisture gradients. Figure 28 compares radiation drying of 0.64-cm. matted wool felt, centrifuged and initially thoroughly wetted, and is typical. The temperature characteristics on this curve are also typical. Although center and surface temperatures of each sample followed different paths, as shown previously, the temperature histories of centrifuged and uncentrifuged samples followed closely similar paths.

Summary

In so far as drying mechanisms are concerned, radiation and convection air drying have been found

to follow the same sequence of events. Thoroughly wetted samples in each case dry at a constant rate which in radiation drying depends upon radiation intensity, surface absorptivity, the temperature, the humidity, and the velocity of the air. In air drying the value of the constant rate depends upon air velocity, temperature, and humidity. After superficial surface water has been evaporated, additional water is caused to flow toward the surface by means of capillary forces. Capillary migration provides the second period of drying and transports to the surface any dissolved matter. The third and final period of drying, involving a diffusion of water vapor, becomes established after the moisture content of the sample has been reduced to a regain which is less than the equilibrium saturation value.

Rate of drying with infrared radiation is proportional to intensity of radiation and is capable of high values compared to convection air drying. The final dry temperature of the sample in radiation drying tends to be high, and rate of change of sample temperature with time, when the sample is almost dry, is very rapid. Close control of final sample temperature and regain is necessary to avoid excessively high temperatures.

There is need for primary radiation intensity standards in the range of high intensities encountered in infrared drying and heating.

In radiation drying, air velocity over the sample has a minor effect upon the rate of drying in all periods, whereas in air drying its effect is major except in the final diffusion period. The effect of changing the temperature of the air is significant in both radiation and air drying. However, neither air temperature nor humidity has as important an effect upon rate in radiation drying as in air drying. In the former, radiation intensity is the predominant variable.

At values of regain greater than 1.2, the rate of radiation drying of wool felt was found to be constant and independent of sample thickness. The effect of sample thickness on drying rate at smaller values of regain was large. Below 0.45 regain, drying rate varied in inverse order of thickness, and drying-rate curve shapes were qualitatively similar to others reported in the literature for air drying.

The effect of radiant transmission, or penetration, becomes increasingly important as the textile becomes drier. In dry or almost dry samples of wool felt it was found that center temperatures may rise considerably (30°C) above surface temperatures because of transmittance. With excessive radiation intensity, purposely imposed, sample cores were found to decompose more rapidly than the surface. In comparative drying tests of air and radiation drying at equal drying rates for thoroughly wet felt samples, it was found that in the low-regain period of drying, the rate in the radiation experiments was considerably greater than in the air experiments. The difference is ascribed to radiant penetration.

Sample color was found to affect the rate of radiation drying. Black-dyed wool felt samples dried more rapidly than white, undyed samples. Differences in drying rate between black and white samples increased as the regain became smaller until, in the final drying period, under a given set of conditions the drying rates of the black samples were about 45 percent higher than those of the white. At equivalent drying rates, black samples were slightly cooler than white samples. Drying rate differences between two black-dyed wool felts with high and low infrared reflectance could be distinguished.

Physical structure of the textile material—that is, whether it is woven, matted, or in loose bulk form—was found to influence rate of drying and values of critical moisture regains between drying periods.

Although characteristic rate-regain drying curves were somewhat different in form for wool felts dried, starting with high (3.5) initial regains and with low (0.30) initial regains obtained by centrifuging, it was found that absolute values of rate, in the region of common regain, did not differ greatly.

Acknowledgment

The authors express appreciation to the Research Advisory Committee of the Textile Research Institute and to Mr. Giles E. Hopkins for helpful suggestions, to Miss Carol F. Bowen for experimental assistance and calculations of data, and to Miss Dorothy F. O'Reilly and Mr. Robert F. Scott for experimental aid. They acknowledge a helpful supply of samples by Mr. W. S. Faurot of the Western Felt Works, Dr. Werner von Bergen of the Forstmann Woolen Company, Mr. W. H. Horne of F. C. Huyck and Sons, and Dr. A. L. Peiker of the Calco Chemical Division of the American Cyanamid Company.

NOMENCLATURE

b = a constant, mm. Hg

h = air-film heat-transfer coefficient, $\frac{\text{g.-cal.}}{\text{m.}^2 \times \text{sec.} \times {}^\circ\text{C}}$ or $\frac{\text{B.t.u.}}{\text{ft.}^2 \times \text{hr.} \times {}^\circ\text{F}}$

I = intensity of radiant flux penetrating surface, $\frac{\text{g.-cal.}}{\text{m.}^2 \times \text{sec.}}$ or $\frac{\text{B.t.u.}}{\text{ft.}^2 \times \text{hr.}}$

I_0 = intensity of radiant flux approaching sample, $\frac{\text{g.-cal.}}{\text{m.}^2 \times \text{sec.}}$ or $\frac{\text{B.t.u.}}{\text{ft.}^2 \times \text{hr.}}$

I_R = intensity of radiant flux reflected from surface, $\frac{\text{g.-cal.}}{\text{m.}^2 \times \text{sec.}}$ or $\frac{\text{B.t.u.}}{\text{ft.}^2 \times \text{hr.}}$

I' = intensity of radiant flux reradiated from surface, $\frac{\text{g.-cal.}}{\text{m.}^2 \times \text{sec.}}$ or $\frac{\text{B.t.u.}}{\text{ft.}^2 \times \text{hr.}}$

k = air-film mass-transfer coefficient, $\frac{\text{g.}}{\text{m.}^2 \times \text{sec.} \times \text{mm. Hg}}$ or $\frac{\text{lb.}}{\text{ft.}^2 \times \text{hr.} \times \text{mm. Hg}}$

L = length of sample in direction of air flow, ft.

m = a constant, $\frac{\text{g.-cal.}}{\text{m.} \times \text{sec.} \times {}^\circ\text{C}}$ or $\frac{\text{B.t.u.}}{\text{ft.}^2 \times \text{hr.} \times {}^\circ\text{F}}$

n = a constant, dimensionless

p_a = partial pressure of water vapor in air, mm. Hg

p_s = water vapor pressure at surface, mm. Hg

r = rate of drying, $\frac{\text{g.}}{\text{m.}^2 \times \text{sec.}}$ or $\frac{\text{lb.}}{\text{ft.}^2 \times \text{hr.}}$

Re = Reynolds number = $\frac{LV_\infty\rho_\infty}{\mu}$, dimensionless

t_a = air temperature, °C or °F

t_s = surface temperature, °C or °F

V = air velocity at a distance from sample, ft./hr.

Λ = latent heat of vaporization, $\frac{\text{g.-cal.}}{\text{g.}}$ or $\frac{\text{B.t.u.}}{\text{lb.}}$

λ = latent heat of vaporization, $\frac{\text{g.-cal.}}{\text{g.-mol.}}$ or $\frac{\text{B.t.u.}}{\text{lb.-mol.}}$

μ = air viscosity at bulk temperature, $\frac{\text{lb. mass}}{\text{ft.} \times \text{hr.}}$

ρ_∞ = air density at a distance from sample, $\frac{\text{lb. mass}}{\text{ft.}^3}$

Regain = weight water per weight dry solids

CONVERSIONS

To convert from centimeters *to* inches, *multiply by* 1/2.54

degrees C	degrees F	1.8, then add 32
$\frac{\text{g.-cal.}}{\text{m.}^2 \times \text{sec.}}$	$\frac{\text{B.t.u.}}{\text{ft.}^2 \times \text{hr.}}$	1.327
grams	pounds	1/454
$\frac{\text{g.}}{\text{m.}^2 \times \text{sec.}}$	$\frac{\text{lb.}}{\text{ft.}^2 \times \text{hr.}}$	0.737
meters/second	feet/second	3.28
millimeters	inches	1/25.4
watts/mm.^{2}	$\frac{\text{g.-cal.}}{\text{m.}^2 \times \text{sec.}}$	2.39×10^5

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(Manuscript received July 14, 1948.)

Dyeing Studies at Elevated Temperatures

Use of Temperatures Between 200°F and 300°F

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Abstract

An apparatus has been built for dyeing textiles in the temperature range of 200°F to 300°F and fundamental dyeing data have been obtained using a wide variety of textile fibers. The high-temperature technique is particularly applicable in the dyeing of those fibers which require a long time for the diffusion of the dye into the individual fibers when it is applied under normal dyeing conditions. The high-temperature technique increases the speed of dyeing to such an extent that dyeings which normally require hours can be produced in a matter of seconds. Commercially acceptable dyeings have been made on wool and wool-cotton unions in less than 90 seconds at 270°F. In general, it can be stated that because of the short periods of contact at these high temperatures the same extent of dyeing can be obtained without any greater change in the tensile characteristics than is obtained when an equivalent dyeing is carried out for a longer time at lower temperatures.

The amount of thermal decomposition of a large number of dyes was determined, and a number which are suitable for high-temperature dyeing have been selected.

A method for the application of dyes by padding and then developing at elevated temperatures has been worked out; this method, owing to the increased speed of dyeing, makes it practical to dye continuously at a rapid rate many fabrics which previously have not been dyed by a continuous process.

IT IS WELL KNOWN that an increase in the dyeing temperature will often aid in promoting levelness, penetration, and exhaustion of dyes. The work of Goodall and Hobday [9, 10] has shown the advantage gained by starting wool dyeings at the boil over the common practice of starting at a low temperature and gradually raising to the boil. In wool dyeing, an increase in the temperature from 200°F to 212°F will cause an increase in the dye penetration and also, with many dyes, added color value. This was shown to be the cause of the difference in color value and penetration between laboratory dyeings made at a vigorous boil and open-kettle dyeings which usually never reach a temperature much higher than 200°F [6]. Figures 1 and 2 show cross sections of such dyeings and illustrate the difference in diffusion into the individual wool fibers. In the dyeing of cellulosic fibers, such as cotton and viscose rayon, an increase in temperature also will cause more rapid diffusion and leveling of dyes, but the amount of dye exhausted is less at the higher temperatures. A recent paper by Boulton [2] emphasizes the advantage of using the highest practical dyeing temperature as a general practice in rayon dyeing.

It has been the established practice to carry out dyeings at temperatures at or below 212°F. In some cases, textile materials have been treated in aqueous solutions in kiers under pressure at temperatures above 212°F, but no references were found in the literature describing dyeings made under these conditions. In fact, Matthews [11] states that wool is destroyed when heated in aqueous solutions at 250°F. Following the concept of continuous dyeing at high temperatures under pressure by one of us [28], the Research Department of the Calco Chemical Division, American Cyanamid Company, began in 1941 a cooperative investigation with the Bachmann Uxbridge Worsted Corporation on a fundamental study of the dyeing of wool and wool-cotton unions at high temperatures under pressure. The Bachmann Uxbridge Worsted Corporation in 1938 began a laboratory development of a method of continuous dyeing at high temperatures under pressure. A continuous-dyeing apparatus was built for this study, the results from which indicated the need for certain fundamental dyeing data in the temperature range of 200°F to 300°F. Sufficient fundamental information has now

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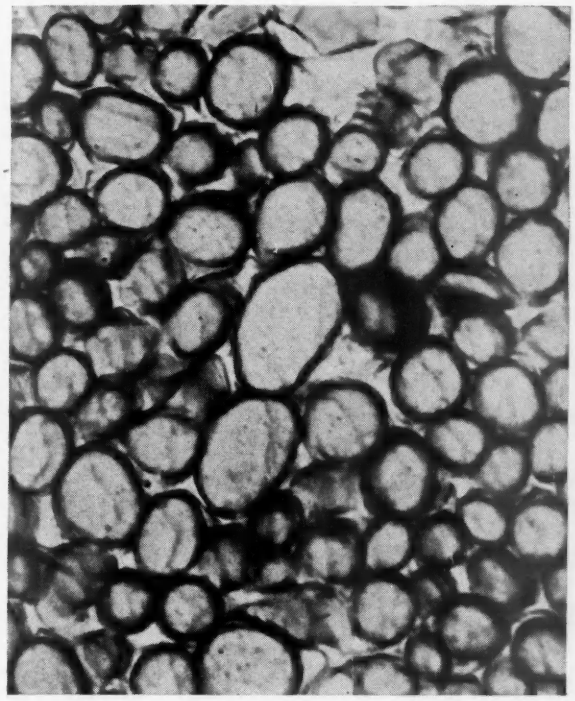

FIG. 1. *Cross section of wool dyed at 200°F.*

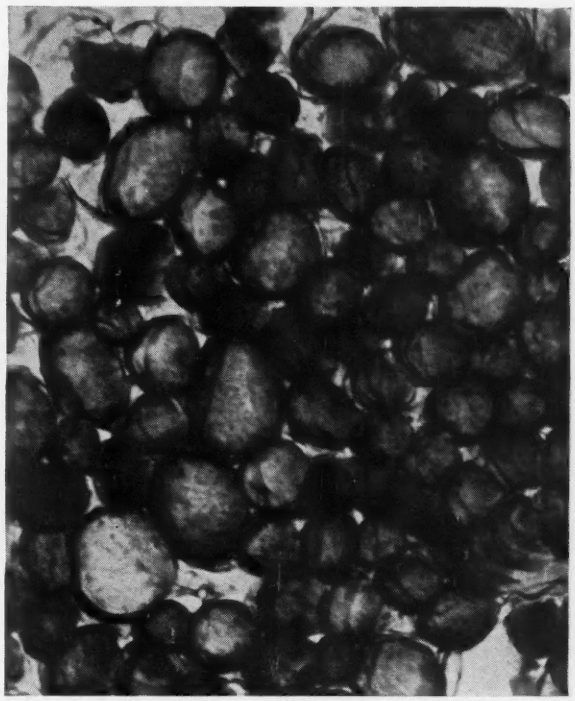

FIG. 2. *Cross section of wool dyed at 212°F.*

FIG. 3. *Cross section of wool dyed at 200°F under 50 lb. per sq. in. pressure.*

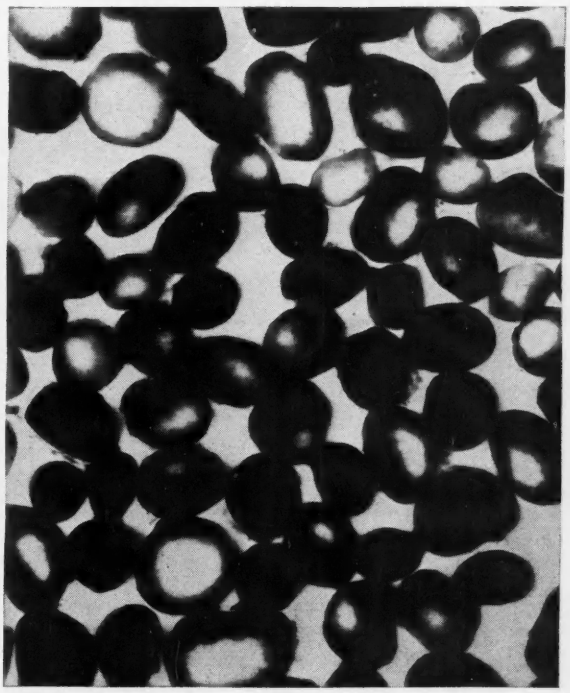

FIG. 4. *Cross section of wool dyed at 200°F under atmospheric pressure.*

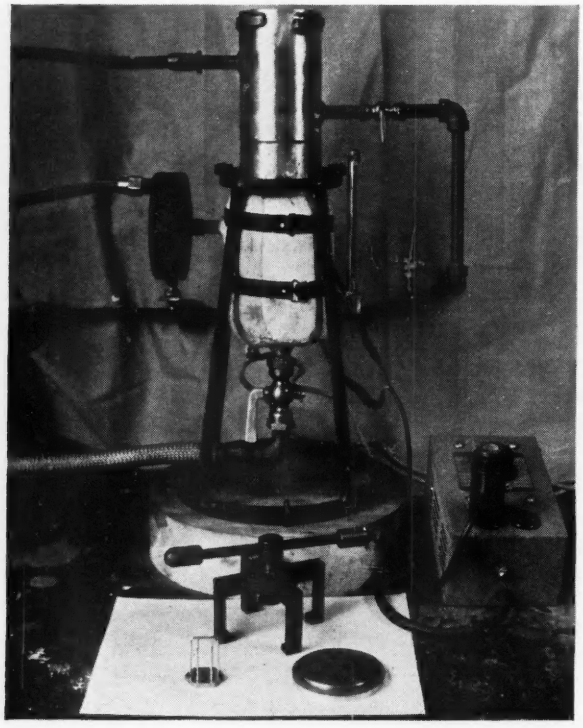

FIG. 5. *Laboratory high-temperature dyeing machine.*

been obtained to warrant publication of some of the factors which are encountered when dyeing is carried out in this temperature range.

Laboratory High-Temperature Dyeing Apparatus

To investigate dyeing in the range of 200°F–300°F, it is necessary to have the system under pressure in order that these high temperatures can be maintained. Experiments have shown that pressure has no effect upon the dyeing process other than permitting the high temperatures to be attained. For example, a dyeing was made at 200°F at atmospheric pressure and then another was made at the same temperature and dyeing conditions but under a pressure of 50 lb. per sq. in. The color values obtained were the same and the penetration and fiber levelness were equivalent, as can be seen in Figures 3 and 4, which show cross sections of the dyeings. A convenient operating pressure of 50 lb. per sq. in. was chosen for all experiments since this pressure permitted temperatures up to 300°F to be attained without boiling. This pressure was maintained by compressed air.

A small laboratory noncontinuous dyeing unit [21] was designed and built at Calco for dyeing at temperatures up to 300°F under 50 lb. per sq. in. air pressure. Figure 5 is a photograph and Figure 6 is a cross-sectional drawing of the unit. As can be seen from the drawing, the apparatus consists of two chambers. In operation, the dye solution or developing bath is placed in the lower chamber. The platform-valve, which separates the two chambers, is pulled into place and then held there by applying 50 lb. air pressure to the lower chamber. The dye solution or developing bath is then heated by electrical coils which surround the lower chamber. The unit is occasionally rocked to agitate the dye bath to obtain a uniform temperature throughout the bath. The sample to be dyed is mounted on a small holder and then placed on the platform-valve which separates the two chambers. When the dye bath has reached the desired temperature, the cover is fastened on the top chamber. Compressed air is then let into the top chamber until the

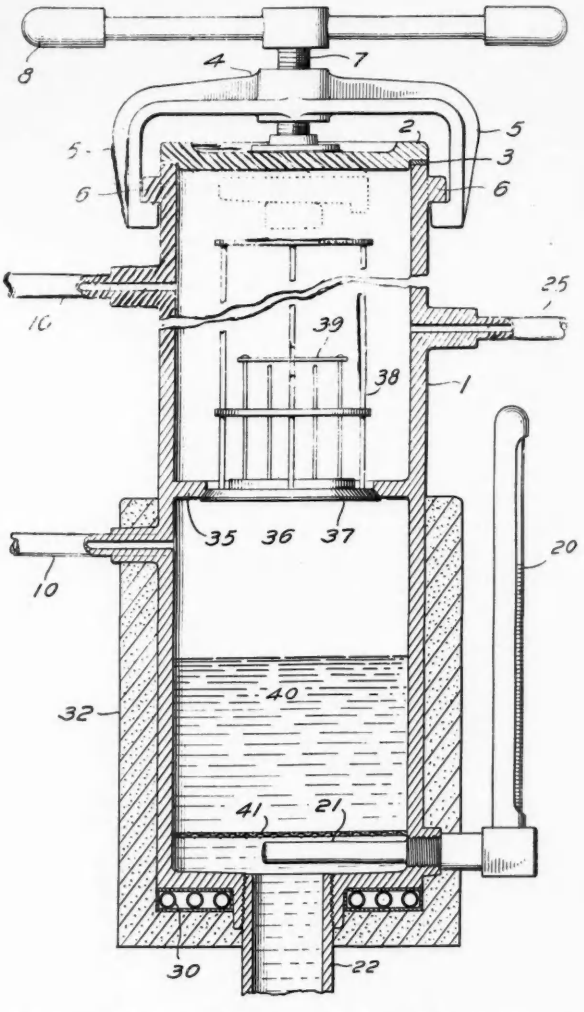

FIG. 6. *Cross-section drawing of high-temperature dyeing machine.*

TABLE I. TENSILE CHARACTERISTICS OF WOOL DYED AT HIGH TEMPERATURES

Dye bath	Temperature of dyeing (°F)	Time of dyeing (sec.)	Tensile strength (lb.)
None (undyed wool)	—	—	2.1
3% solution Calcocid Wool Blue B (C.I. 209)	290	34	1.7
3% solution Calcocid Wool Blue B (C.I. 209)	280	40	1.8
3% solution Calcocid Wool Blue B (C.I. 209)	270	50	2.0
3% solution Calcocid Wool Blue B (C.I. 209)	260	60	2.0
3% solution Calcocid Wool Blue B (C.I. 209)	250	100	2.2
3% solution Calcocid Wool Blue B (C.I. 209)	240	140	2.1
3% solution Calcocid Wool Blue B (C.I. 209)	230	220	2.1
3% solution Calcocid Wool Blue B (C.I. 209)	220	300	2.1
3% solution Calcocid Wool Blue B (C.I. 209)	210	480	2.2

pressure in the upper chamber equals that in the lower. When the pressure in the two chambers is equal, the platform-valve drops, permitting the sample to fall into the dye bath. The sample is agitated during the dyeing period by rocking the unit. When the sample has dyed for the desired length of time, the dye bath is blown out of the lower chamber by opening the valve at the bottom, and the dyed sample is immediately washed by allowing cold water to enter through the valve attached to the top chamber. This method and apparatus allow a sample to be entered into a high-temperature bath without preheating or steaming and to be removed from the bath quickly and cooled. Thus it is possible to have very short periods of immersion in the high-temperature bath, which would simulate a continuous operation. The above method and apparatus, if used on a large scale, would obviously afford a batch process for commercial high-temperature dyeing.

Tensile Characteristics of the Dyed Fibers

Dyeings made at elevated temperatures for short periods resulted in no apparent damage to the wool. However, in view of Matthews' [11] statement that wool disintegrated in aqueous solution at 250°F, experiments were performed to determine the tensile characteristics of wool dyed at various temperatures and times. Skeins were dyed in a 3% solution of Calcocid Wool Blue B (C.I. 209) in the laboratory apparatus at the temperatures and for the times given in Table I. Under these conditions, all of the samples gave approximately the same depth of shade. It is evident from the tensile-strength data, also shown in Table I, that dyeing at temperatures up to 270°F for the times indicated causes no loss in tensile strength, within experimental error. At 280°F and 290°F, there is some loss in tensile strength. Table II gives data comparing dyeings made at elevated temperatures with those of similar shades prepared by regular dyeing methods. Here it is evident that there is no significant tensile-strength loss due to dyeing at high temperatures.

Single-fiber stress-strain data obtained to check the results on the wool skeins are shown in Table III. The 30% index [5, 23] was used to correlate the results. The 30% index is the energy required to stretch a wet fiber to 30% elongation after a treatment divided by the energy requirement prior to the

TABLE II. TENSILE CHARACTERISTICS OF WOOL DYED AT HIGH TEMPERATURES VS. REGULAR TEMPERATURES

Dye bath	Temperature of dyeing (°F)	Time of dyeing (sec.)	Tensile strength (lb.)
None (untreated control)	—	—	2.17
Calcocid Wool Blue B (C.I. 209)	212	3,600	2.10
Water only	212	3,600	2.10
Water only	230	110	2.08
Calcocid Wool Blue B (C.I. 209)	230	110	2.21
Water only	290	110	2.05
Calcocid Wool Blue B (C.I. 209)	290	110	2.08
Calcocid Wool Blue B (C.I. 209)	290	15	2.27

TABLE III. STRESS-STRAIN DATA ON DYED SINGLE FIBERS OF WOOL

Dye bath	Time of dyeing	Temperature of dyeing	30% index
3% solution Calcocid Orange Y (C.I. 151)	60 sec.	270°F	0.98
3% solution Calcocid Green S (C.I. 737)	60 sec.	270°F	0.91
10% solution $NaH_2PO_4 \cdot H_2O$	60 sec.	270°F	0.92
Calcocid Green S, 10% dyeing	1 hr.	at boil	0.93
Calcocid Green S, 1% dyeing	1 hr.	at boil	0.88
Calcocid Orange Y, 10% dyeing	1 hr.	at boil	1.02
Calcocid Orange Y, 1% dyeing	1 hr.	at boil	0.95

TABLE IV. TENSILE CHARACTERISTICS OF FIBERS DYED AT 270°F

		Tensile strength (lb.) Before dyeing	After dyeing
Cotton unbleached sheeting	warp	40	47
	filling	45	45
Viscose	warp	106	111
	filling	79	76
Nylon	warp	98	109
	filling	88	104
Acetate	warp	60	13
	filling	28	14

treatment. The smaller the values in Table III, the more the fibers have been modified. These measurements also showed that the tensile characteristics of wool dyed at elevated temperatures were similar to those of wool dyed by the normal dyeing method. There was some evidence of a difference in effect between different dyes, but this was true both at normal and at elevated temperatures.

The data in Tables I, II, and III were obtained from tests made on wool because it was thought to be the fiber most readily affected by elevated temperature. Table IV shows the effect of high-temperature treatment on the tensile characteristics of several other textile fibers. For many years cotton has been subjected to kier boiling at high temperatures and there is no obvious reason why elevated temperatures for dyeing should have bad effects upon its tensile characteristics. In the dyeing of nylon, it is obvious that the temperature conditions must be lower than those used for setting, and in our experiments nylon has not offered any observed practical difficulty. Acrylonitrile fibers also have been treated with no apparent loss in tensile characteristics. The results show that the high temperature had a damaging effect on the acetate fiber.

In general, it can be stated that, because of the short times of contact at the high temperatures, the same extent of dyeing can be obtained without greater damage than is obtained for the same reaction during a longer dyeing time at low temperatures. In other words, some change in tensile characteristics may result because of the reaction of dye with the fiber, and whether this reaction takes place at high or at low temperatures, the same extent of reaction will cause the same damage. The work of Carlene, Fern, and Vickerstaff [7] on nylon and Skinner and Vickerstaff [22] and Goodall and Hobday [10] on wool indicates that under certain conditions there is a reaction with excessive amounts of dye which causes a breakdown of the fiber with an accompanying loss in tensile strength. High temperatures will greatly increase the rates of these reactions and, therefore, the exposure of the fibers to these conditions must be limited.

TABLE V. THERMAL DECOMPOSITION OF DYES AFTER 8 HOURS AT 260°F

Name of dye	C.I. No.	% decomposition
Calcocid Alizarine Blue SKY	1088	<10
Calcocid Milling Red RC Conc.	430	<10
Calcofast Wool Orange RN	—	<10
Calcomine Brilliant Yellow Conc.	365	<10
Calcodur Orange EGL	Pr. 72	<10
Calcocid Milling Red G	443	10–20
Calcocid Violet 4BXN	698	10–20
Calcodur Blue SL Conc.	Pr. 71	10–20
Calcodur Brown BRL	—	10–20
Calcocid Fast Blue SR	208	20–50
Calcocid Milling Yellow 3G	Pr. 642	20–50
Calcomine Navy R	—	20–50
Calcomine Fast Red 8B	—	20–50
Calcocid Fast Black B	307	>50
Calcocid Milling Red 3R Conc.	275	>50
Calcodur Blue 4GL	533	>50
Calcodur Red 8BL	278	>50

Thermal Decomposition of Dyes

A disadvantage of continuous dyeing of textile materials at high temperatures is the thermal decomposition of the dyes when held at elevated temperatures over a period of several hours. The amount of thermal decomposition of a large number of acid and direct dyes in 0.5% concentrations has been deter-

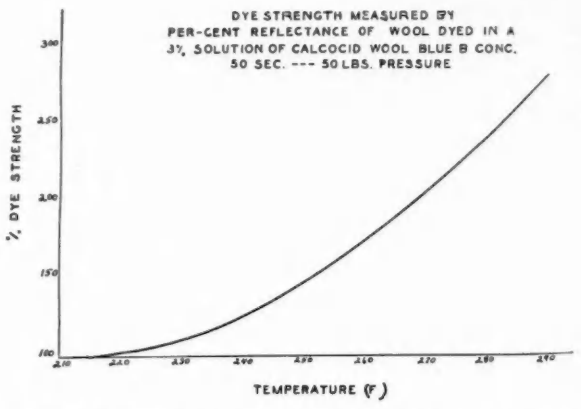
DYE STRENGTH MEASURED BY
PER-CENT REFLECTANCE OF WOOL DYED IN A
3% SOLUTION OF CALCOCID WOOL BLUE B CONC.
50 SEC. --- 50 LBS. PRESSURE
% DYE STRENGTH
TEMPERATURE (F)

FIG. 7. *Dye color strength of wool vs. temperature of dyeing.*

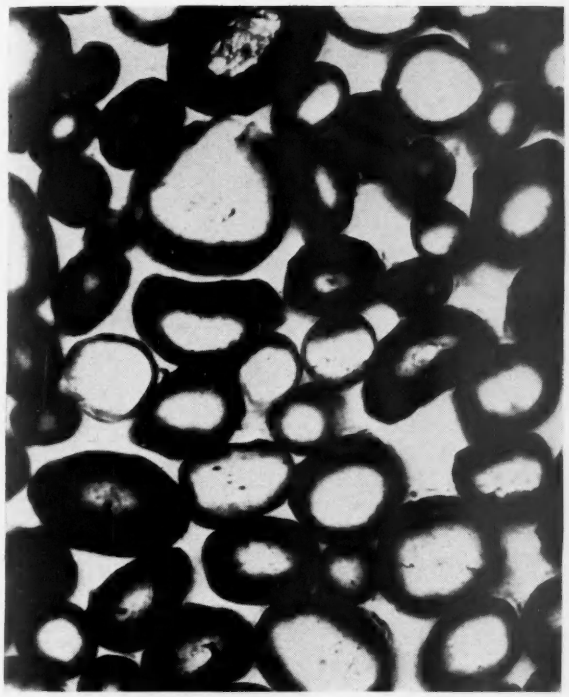

FIG. 8. *Cross section of wool dyed at 210°F.*

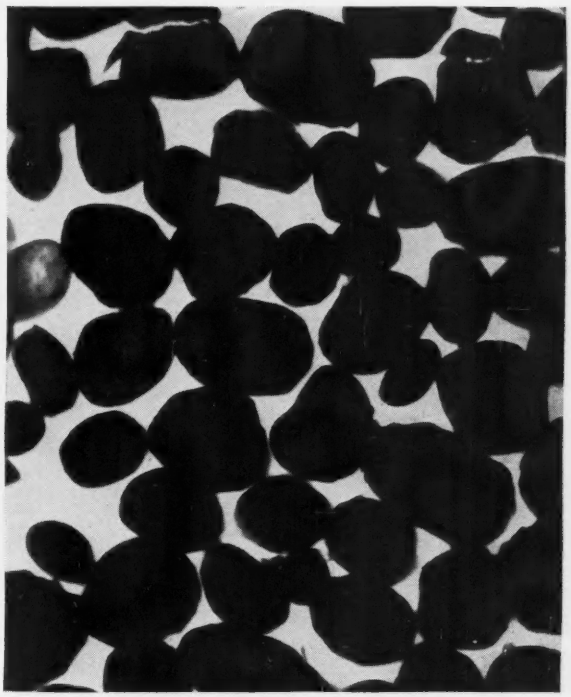

FIG. 9. *Cross section of wool dyed at 290°F.*

mined. Table V shows a few typical examples of the results that were obtained.

These tests were made by heating solutions of the dyes at 260°F and 30 lb. per sq. in. pressure for 8 hours. Dye color-strength determinations were made on the solutions by measurements in the spectrophotometer. Since these tests were carried out in a static condition in an autoclave, it is not necessarily true that the same decomposition would take place under a dynamic condition where dye would be continuously added and removed, but the tendency would still be present. Furthermore, the textile fibers may have an effect upon the decomposition of the dyes. An attempt was made to determine this by inserting small amounts of wool and cotton into concentrated dye solutions, but there was no definite indication that they affected the decomposition. No attempt was made to determine the rate of decomposition.

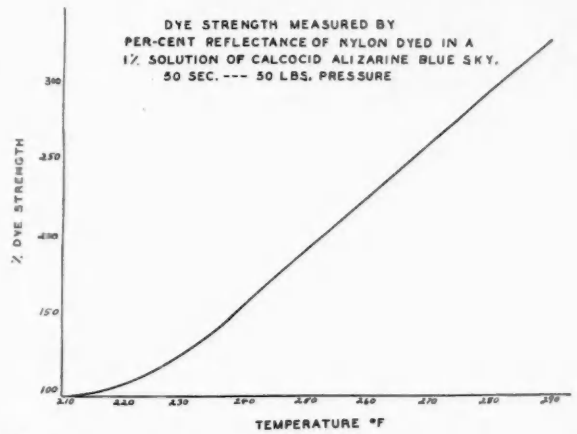
DYE STRENGTH MEASURED BY
PER-CENT REFLECTANCE OF NYLON DYED IN A
1% SOLUTION OF CALCOCID ALIZARINE BLUE SKY.
50 SEC. --- 50 LBS. PRESSURE
% DYE STRENGTH
TEMPERATURE °F

FIG. 10. *Dye color strength of nylon vs. temperature of dyeing.*

Dyeing from Concentrated Dye Baths

The method used in the early work on high-temperature dyeing consisted of passing the undyed cloth through a concentrated dye solution at an elevated temperature under pressure for a short time. The section of the paper immediately following covers some of the data obtained from dyeings made by this method. In contrast to the early method for dyeing at high temperatures, a modified method was developed. This modified method consisted of first padding the cloth with an aqueous solution of dye at normal temperatures and then developing the padded cloth in an aqueous bath at elevated temperatures. This method, with some of its advantages, will be discussed later in this paper.

Color Value vs. Temperature

According to nearly all of the current theories, dyeing takes place by the diffusion of the dye molecules

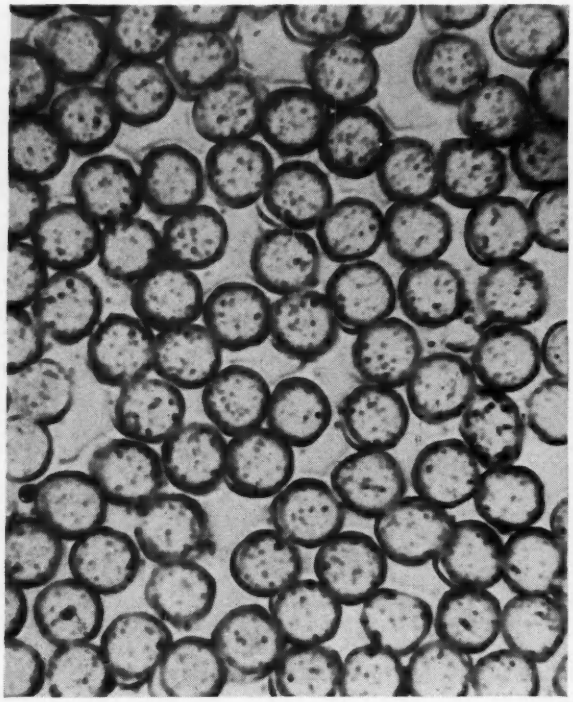

FIG. 11. *Cross section of nylon dyed at 210°F.*

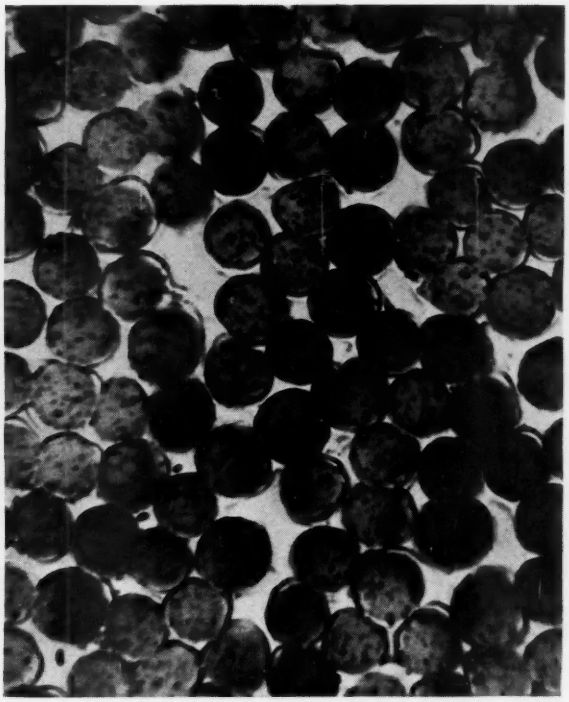

FIG. 12. *Cross section of nylon dyed at 290°F.*

along intermicellar spaces. In other words, on placing the fiber in the dye bath, swelling takes place, resulting in areas through which dye can diffuse. Speakman, Stott, and Chang [24] have shown, experimentally, that the swelling of a wool fiber in water increases with rise of temperature above 105°F. It is well known that the rate of diffusion is increased by a rise in temperature and is decreased as the size of the molecule becomes larger. Most acid and direct dyes are salts of dye acids. These dyes may exist in solution as ions, as molecular dispersion, or as colloidal dispersions. The work of Valko [26, 27] and of Robinson and his collaborators [13, 16–19] has shown that few dyes are dispersed in aqueous solutions as single molecules, although many probably exist as simple aggregates of 2 to 10 molecules. Many others have aggregation numbers of 100 or higher. Depending upon the degree of aggregation, the actual size of the dye particle in solution will vary, and this, in turn, will determine whether it is possible for the dye to enter the fiber by way of the intermicellar spaces. It has been found, as reported by Valko, that the degree of aggregation varies with temperature; hence it is possible to have a colloidal solution at room temperature which changes to a semi-colloidal or nearly true solution at higher temperatures. The diameter of the capillary pores * in viscose cellulose, calculated from measurements of permeability to water, is of the same order as the length of a typical direct cotton dye molecule [12, 15]. Since it is thought that these pores are responsible for the transport of dye to the interior of the fiber, it is almost certain that the dyes are absorbed molecu-

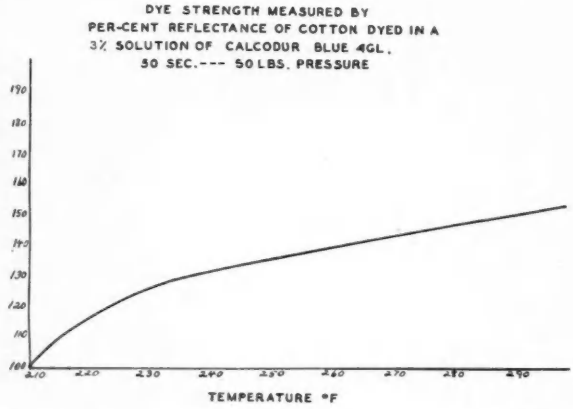

FIG. 13. *Dye color strength of cotton vs. temperature of dyeing.*

* The word "pore" is used here as in the referred literature. However, the authors have the conception of this as a network of cellulose chains which act like pores but are not necessarily continuous or like capillary tubes of a known cross section.

FIG. 14. *Cross section of cotton dyed at 210°F.*

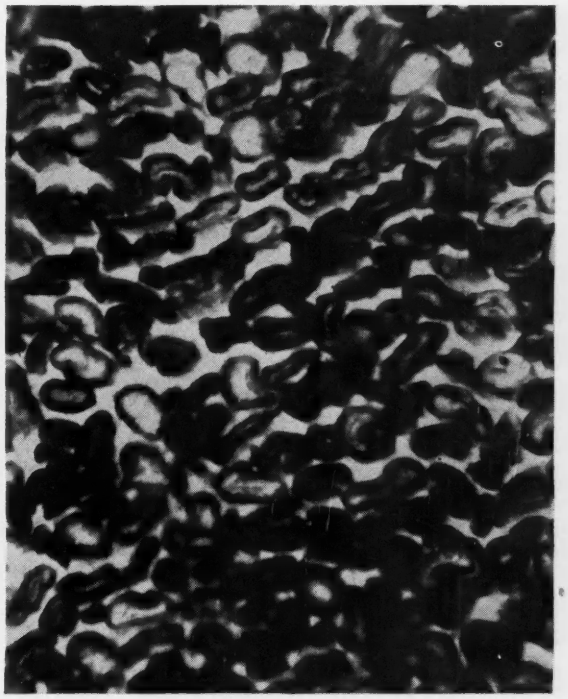

FIG. 15. *Cross section of cotton dyed at 290°F.*

larly and not as dye micelles [15, 25]. Thus, it is evident that high temperatures promote dyeing by increasing the pore size, decreasing the aggregation of the dye, and increasing the rate of diffusion.

In Figure 7, the dye color strength, measured by percent reflectance, of wool samples dyed for 50 sec. in a 3% solution of Calcocid Wool Blue B is shown plotted against the temperature of dyeing. From this curve, it can be seen that the dye color strength of the wool increased from 100% to 280% as the dyeing temperature increased from 210°F to 290°F. Figures 8 and 9 show the cross sections of the dyeings made at 210°F and 290°F. In the short time of dyeing, the one at 210°F shows poor penetration and uneven dyeing from fiber to fiber. The dyeing made at 290°F shows superior penetration and fiber levelness. In like manner, it can be seen from Figure 10 that when nylon was dyed in a 1% solution of Calcocid Alizarine Blue SKY (C.I. 1088), the dye color strength increased from 100% to 300% as the temperature of the dye bath increased from 210°F to 290°F. Figures 11 and 12 show cross sections of the 210°F and 290°F dyeings. At the low temperature, the dyeing was a very light blue and, as can be seen in Figure 11, the dye had just started to penetrate into the individual fibers. At the high temperature, the dyeing was a dark blue and the dye had penetrated through the entire fiber. Cellulosic materials did not show so great an increase in color value with an increase in temperature as did the protein materials. Figure 13 shows that the color value of cotton, dyed in a 3% solution of Calcodur Blue 4GL (C.I. 533), was increased only from 100% to 150% as the temperature increased from 210°F to 290°F. Figures 14 and 15 show cross sections of cotton dyed at 210°F and 290°F, respectively. Viscose rayon, dyed under the same conditions as the cotton, showed

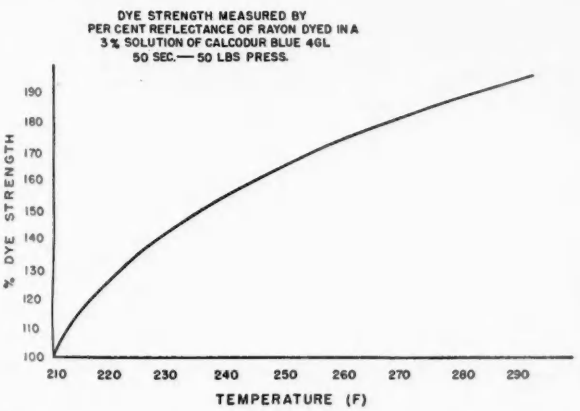

FIG. 16. *Dye color strength of viscose vs. temperature of dyeing.*

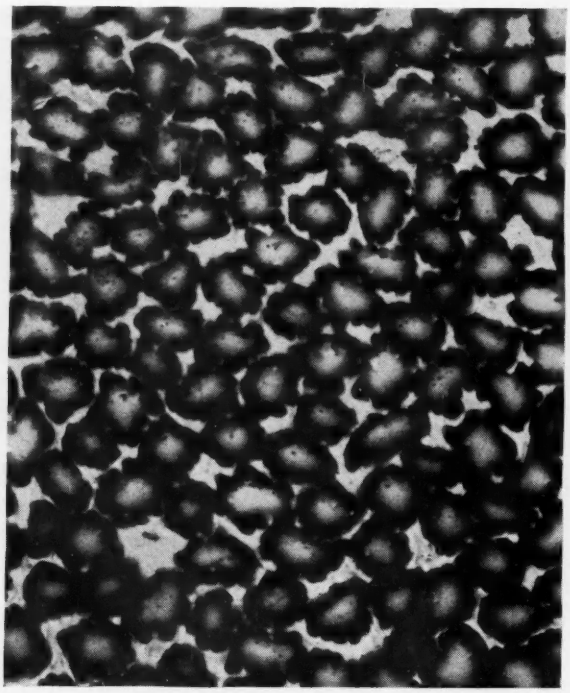

FIG. 17. *Cross section of viscose dyed at 210°F.*

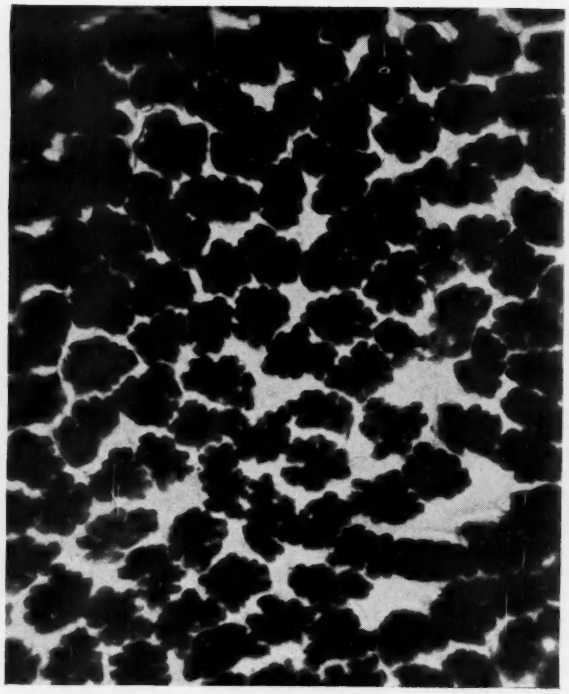

FIG. 18. *Cross section of viscose dyed at 290°F.*

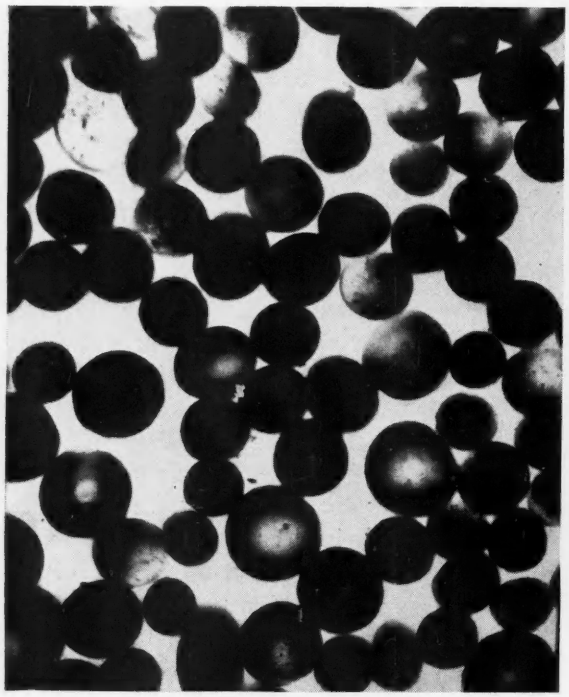

FIG. 19. *Cross section of Aralac dyed at 210°F.*

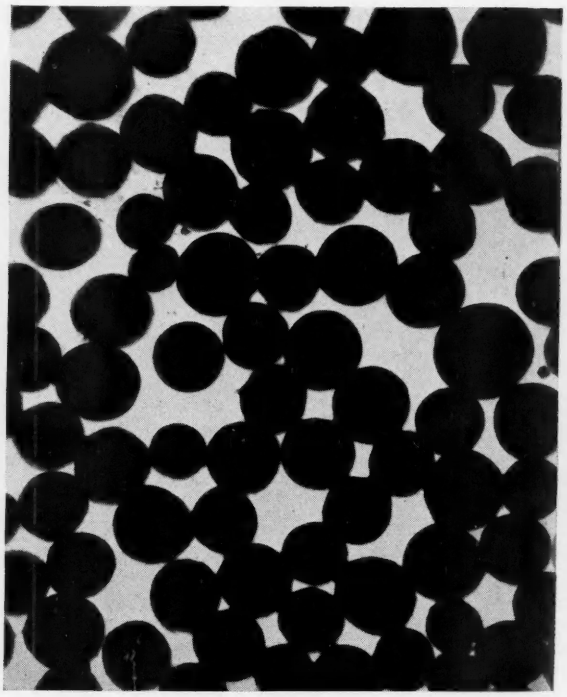

FIG. 20. *Cross section of Aralac dyed at 290°F.*

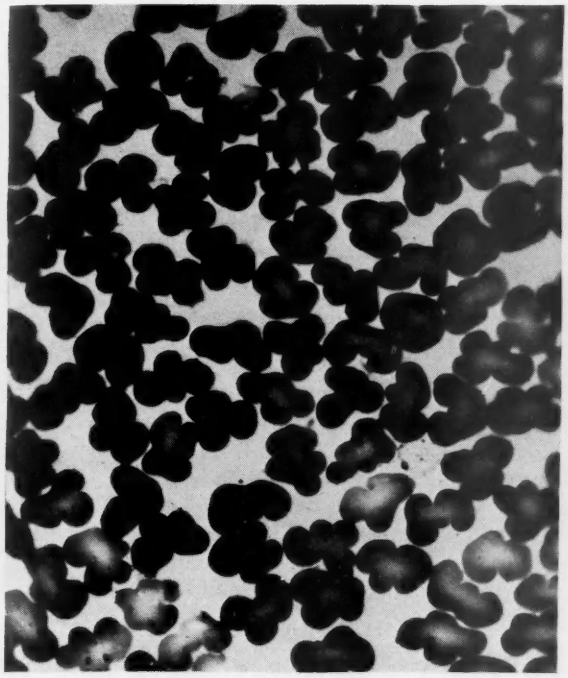

FIG. 21. *Cross section of acetate rayon dyed at 210°F.*

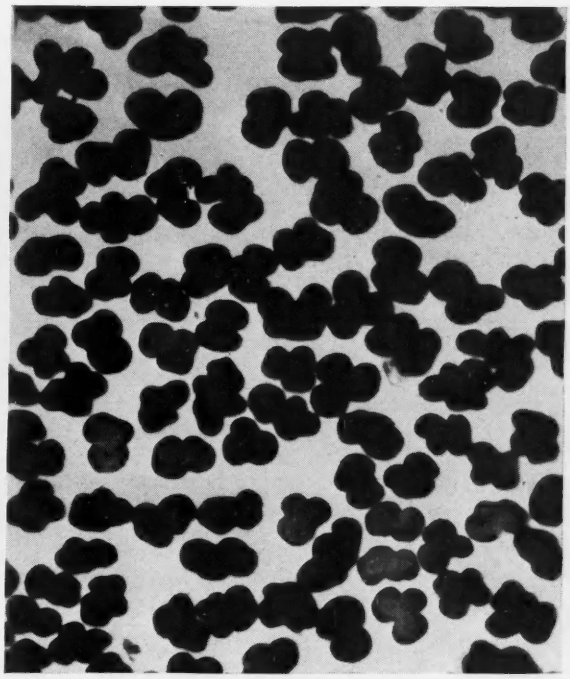

FIG. 22. *Cross section of acetate rayon dyed at 290°F.*

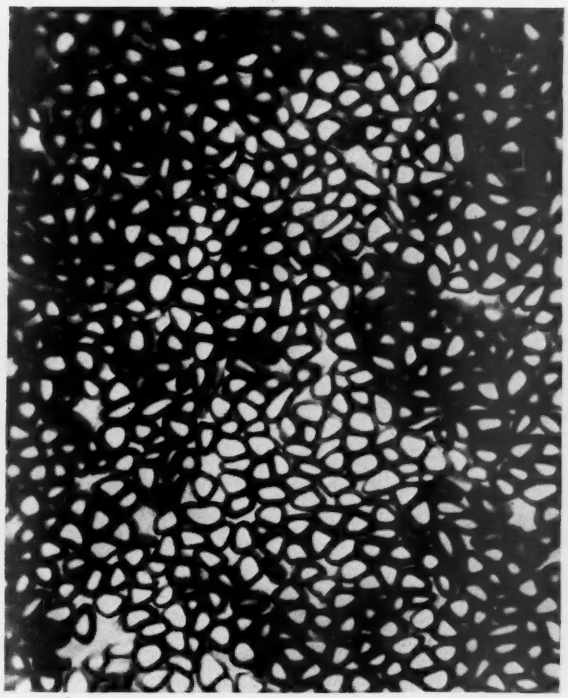

FIG. 23. *Cross section of silk dyed at 210°F.*

FIG. 24. *Cross section of silk dyed at 290°F.*

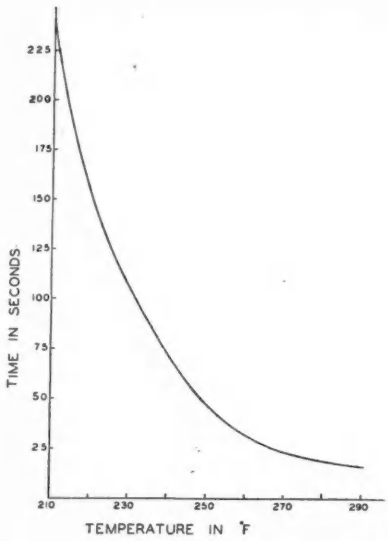

FIG. 25. *Time vs. temperature required to dye wool to the same shade.*

an increase from 100% to 195%. This is shown in Figure 16. Figures 17 and 18 show cross sections of viscose dyed at the two temperatures, 210°F and 290°F. This was a greater increase than was obtained with cotton but much less than that obtained with wool or nylon. Cross sections of dyeings of Aralac, cellulose acetate, and silk made at 210°F and 290°F for 50 sec. are shown in Figures 19–24.

Other things being equal, the amount of dye taken up by a fiber at equilibrium usually decreases as the temperature is increased, but the rate of dyeing increases [3, 8, 20]. The rate of dyeing of protein fibers is increased far more by a temperature increase than that of cellulosic fibers. This is no doubt due to the necessity for more swelling in wool fibers before diffusion can take place than is the case for cellulosic fibers. Diffusion of direct dyes into cellulose, in most cases, is very rapid even at low temperatures.

Color Value vs. Time of Dyeing

Figure 25 shows that the time required to dye wool to the same depth of shade, using a 3% solution of Calcocid Wool Blue B Conc. at 290°F, is only one twenty-eighth of the time required at 210°F. At a temperature of 270°F, dyeings of wool and wool-cotton unions, that were commercially acceptable, have been made in less than 90 seconds. Figure 26 shows how the dye color strength of cotton varies with time of dyeing. All of these experiments were

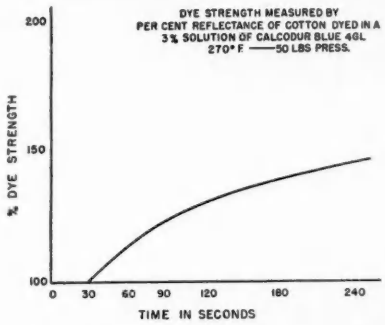

FIG. 26. *Dye strength of cotton vs. time of dyeing.*

made at 270°F using a 3% solution of Calcodur Blue 4GL. The color strengths of the dyed samples were measured by reflectance with a spectrophotometer.

Effect of pH in High-Temperature Dyeing

The importance of pH control in wool processing has been pointed out recently by Mooradian and Millson [14] and by the Philadelphia Section of the American Association of Textile Chemists and Colorists [1]. The effect of pH upon dyeing varies with both the type of fiber and the type of dye used. According to current theories, when wool is placed in an acid solution, hydrogen ions diffuse rapidly into the fiber and neutralize the charges on the carboxylate groups. Because of their small size, these hydrogen ions may even enter the crystalline portions of the fiber. This causes the fiber to become charged positively and to attract anions. These anions (dye ions, sulfate ions, etc.) diffuse into the fiber through the intermicellar spaces and approach as closely as possible to the ionized amino groups of the protein. The anions can move very close to the substituted ammonium ions in the main amorphous portion of the fiber, and the formation of a salt between the dye anions and the ammonium ions of the protein probably occurs. Skinner and Vickerstaff [22] have shown that the dyeing mechanism in the case of nylon appears to be similar to that of wool. Thus, with few exceptions, the dyeing of protein fibers is usually carried out under acid conditions. It is generally agreed that the dyeing of cellulosic materials by direct dyes takes place by means of some type of hydrogen bonding [4], and therefore the pH of the dye bath has little effect upon the amount of dye taken up by the cellulosic fiber.

When acid dyes are applied to protein fibers at high temperatures, it is necessary to control the pH since

this factor has a marked influence on the rate of dye take-up. Low pH values result in rapid exhaustion, but they must be avoided because the dye deposits too rapidly on the outer surface of the fibers. Since the time of dyeing is so short, there is little opportunity for leveling to take place and, when rapid exhaustion occurs, unevenness and poor penetration result. Low pH conditions also tend to make many dyes less soluble and thus present more problems in application.

Wool and other protein fibers are damaged by alkali. Experiments in the high-temperature dyeing machine have shown that this effect is accelerated by an increase in the temperature. It was found that wool could be dyed with no apparent damage at pH's as high as 6, but in continuous dyeing, where the material is under tension, the pH should be kept at 5 or below. The best wool dyeings were obtained at a pH of 4.

It should be remembered that the concentration of dye being used in these experiments is many times stronger than that used in normal dyeing procedures. As a result, any diluent, such as NaCl or Na_2CO_3, present in the dye for purposes of standardization will be present in the dye bath in much higher concentrations than usual. This affects the pH of the bath, the solubility of the dye, and the dyeing process.

In general, it has been found that the pH should be as high as is possible but low enough not to cause damage under the conditions of temperature and time of immersion. Under these conditions, the exhaustion of the dye will be slower but the diffusion will be more rapid, and better penetration and distribution of dye will be obtained.

Effect of Dye Concentration

In order to obtain heavy shades in the dyeing of wool or wool-cotton unions by the high-temperature dyeing method, it is necessary to use relatively high concentrations of dye because of the short time of dyeing. These high concentrations produce rapid diffusion of the necessary amount of dye from the bath into the center of the fiber in the short time allowed for dyeing. Relatively constant dye concentration in the dye bath is considered necessary in order to obtain uniform shades in continuous dyeing. However, such a small amount of dye is exhausted from this bath in comparison to its concentration that considerable dye can be removed before a shade change is observable. It is believed also that these high concentrations of dye may have some effect upon the leveling and penetration.

Dyeing by Padding and Developing

In the course of the investigation, it became apparent that there were many disadvantages in dyeing continuously from a concentrated dye bath. To overcome some of these disadvantages, a modified method was developed which consisted of first padding the cloth with an aqueous solution of dye at normal (below 212°F) temperatures and then developing the padded cloth in an aqueous bath at elevated temperatures maintained under pressure. This modified method has the following advantages:

1. Padding offers another method of controlling the shade and obtaining a uniform deposition of dye over the cloth, in contrast to trying to control the exhaustion of several dyes in a large tank of concentrated dye solution, and is especially convenient if the dyes exhaust at markedly different rates.
2. There is less thermal decomposition of the dye. In dyeing from a concentrated dye bath, the dye remains at an elevated temperature for many hours. In dyeing by the padding-developing method, the dye is brought into the high-temperature machine by the cloth, held at the elevated temperature for a very short time, and then removed by the cloth. This makes it possible to use many dyes that would be too unstable to use by the original method of dyeing.
3. It is possible to dye smaller lots profitably, as there is no large concentrated dye bath to discard at the end of the run.
4. The shade of dyed cloth is brighter, and better penetration is obtained in some cases. In dyeing from concentrated baths, thermal decomposition may produce a dulling of shade with many dyes, and in these cases it is impossible to produce the true bright shade.

Padding

Some leveling occurs during the developing of the padded cloth, but to obtain a level dyeing the padding must be even and uniform. Generally speaking, the depth of shade is controlled by the pick-up and the concentration of the padding solution. For dyeing heavy shades by this method, very concentrated padding solutions were required, especially in the case of wool dyeing. In padding with acid and direct dyes, it was found that the best results were obtained when the dye was completely in solution. It thus be-

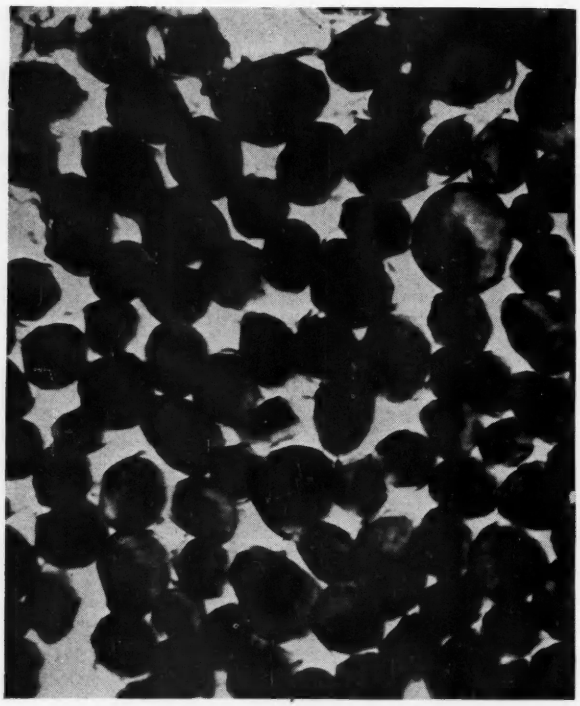

FIG. 27. *Cross section of wool dyed at 275°F for 1 minute.*

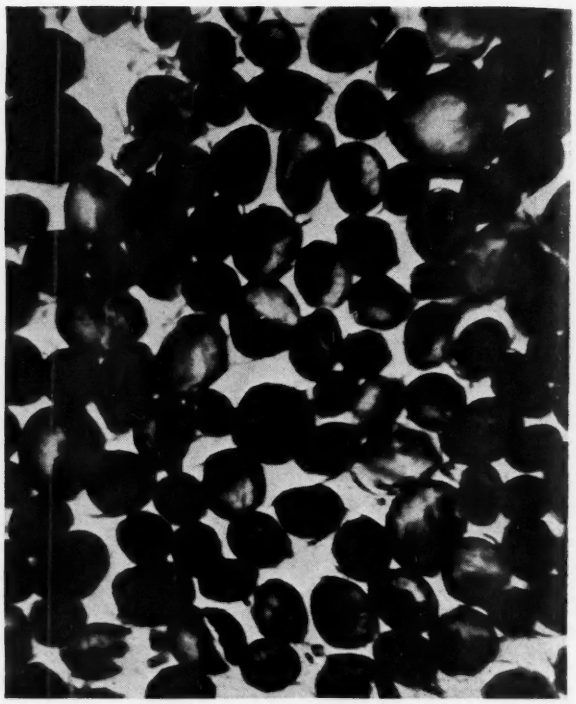

FIG. 28. *Cross section of wool dyed at 200°F for 120 minutes.*

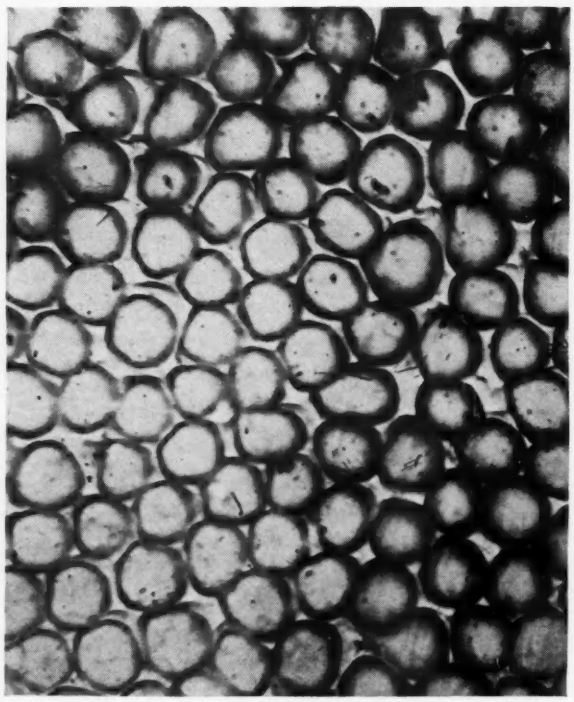

FIG. 29. *Cross section of nylon dyed at 275°F for 75 seconds.*

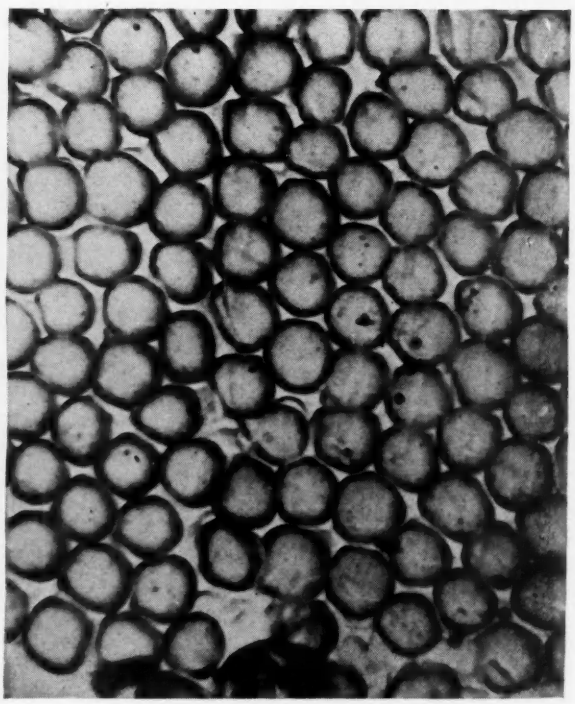

FIG. 30. *Cross section of nylon dyed at 212°F for 1 hour.*

came necessary to determine the solubility of acid and direct dyes and also to find methods for increasing their solubility. A method of determining dye solubilities at a specified temperature was developed and published recently [29], together with the solubility in water of a number of acid and direct dyes.

It was found that regular cotton padding techniques could not be used in padding wool. Therefore, an extended study was made showing the effect of different variables upon the padding of cotton, wool, and rayon. These variables consisted of chemical and physical constitution of the cloth; addition of wetting agents to the cloth and bath; variations in the pH, temperature, viscosity, and composition of the dye bath; number of dips and nips; time of immersion; speed of padding; and pressure between the squeeze rolls. The results of this study will appear in a future publication.

Developing

When the padded cloth passes into the developing bath, a number of things occur simultaneously. Part of the dye is washed off the padded cloth by the developing bath. The textile fiber starts to swell and the dye starts to diffuse into the fibers. At the same time, dye in the developing bath begins to dye the fibers. Experiments have shown that the requirements for a developing bath are: (1) it should reduce bleeding from the padded cloth into the bath to a minimum; (2) the pH of the bath should be adjusted to promote dyeing and prevent damage to the fibers; (3) the bath should promote level dyeing; and (4) the ingredients of the bath should be relatively inexpensive. A number of developing baths of varying composition were found which fit the above requirements. One of the best of these was an acetate buffer of pH 4 to 4.5.

In a continuous process, equilibrium conditions are established in the developing bath so that while the padded cloth is passing through the developing solution the dye bleeding off is equal to the amount dyeing back on the cloth. One of the major problems in continuous dyeing is the determination of the amount of dye to be added to the developing bath at the beginning to maintain the required equilibrium. The concentration of dye necessary in the developing bath for equilibrium has been found to depend upon the dye used, the strength of the padding, the time of immersion, the temperature of the developing bath, the cloth-developing bath ratio, and the composition of the developing bath. All these factors are known to have an effect upon continuous dyeing at temperatures below the boil and obviously had to be taken into consideration at elevated temperatures.

The pH of the developing bath is critical, especially when dyeing protein fibers. A variation in pH does not produce so great a change in dyeing cellulosic fibers. As the developing bath becomes more acid, there is less bleeding of dye from the padded wool samples. However, when the pH becomes too low, the dye is precipitated on the outside of the fiber, which causes an uneven, poorly penetrated dyeing. Since the pH is critical in wool dyeing, a buffered developing bath permits easier control because it resists changes in pH.

The addition of electrolytes, such as sodium chloride and sodium sulfate, to the developing bath has an entirely different effect upon protein-fiber dyeing from that which it has upon cellulosic-fiber dyeing. A developing bath containing a low concentration of salt in contrast to one containing a high concentration of salt will cause better exhaustion in wool dyeing, giving very little bleed-off, but it will lower the exhaustion in cotton dyeing and cause a large bleed-off. It is therefore evident that the quantity and kind of electrolyte in the developing bath will affect the exhaustion and diffusion properties. The choice of the quantity of salt will depend, as in normal dyeing, upon the fibers being dyed and the dyes used in the dyeing.

Experiments also have been carried out in which attempts were made to develop the padded samples by steaming at the high temperatures equivalent to those used in the liquid developing baths. Even though saturated steam atmospheres were present, the results with respect to penetration and color strength were not so good as when the liquid developing bath was used. These results would indicate that high temperature is not the only factor, and that the liquid dyeing or developing bath influences the penetration and the color value obtained.

Application of High-Temperature Technique

As can be seen from the above discussion, the high-temperature technique is particularly applicable in the dyeing of those fibers which require a long time for the diffusion of the dye into the individual fibers when it is applied under normal dyeing conditions. For example, worsted cloth containing an acetate effect thread was padded with a 10% solution of

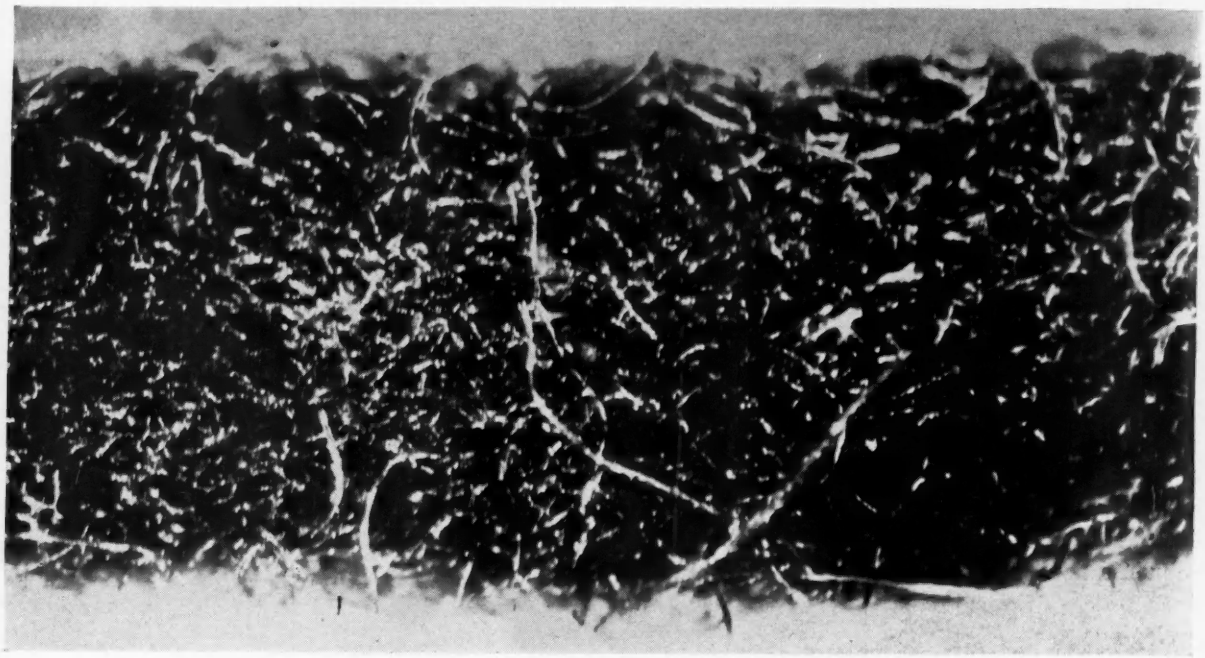

FIG. 31. *A section of hat felt cut to show complete penetration of dye.*

Calcocid Indo Blue B. Part of this material was developed at 275°F for 1 minute in a pH 4 acetate buffer and another part was developed in an open dye kettle with 10% Glauber's salt and 4% acetic acid (28%) of 200°F for 2 hours. Both of the dyed samples were level, well-penetrated dyeings. Photomicrographs of cross sections of these dyeings are shown in Figures 27 and 28. The acetate effect thread was much whiter in the case of the high-temperature dyeing. The light, crock, wash, and fulling tests showed no difference in fastness between the high-temperature dyeing and the one at 200°F. It is therefore evident that satisfactory dyeings can be obtained in 60 seconds by the high-temperature method which are equal or superior to those made by the regular commercial procedure which requires several hours.

Olive drab shades have been obtained on nylon by padding with a solution of Calcofast Wool Blue 2G, Calcofast Wool Orange RN, and Calcofast Wool Yellow R, and then developing for 75 seconds at 275°F in a sulfuric acid bath (¼ oz./gal.). The penetration was better in this dyeing than in one made by boiling the same padded material with 8% sulfuric acid (on weight of nylon) for 1 hour, but neither showed complete penetration of the dyes to the center of the fibers. Figures 29 and 30 show cross sections of these dyeings. Although the high-temperature dyeing was superior in penetration to the dyeing made at the boil, both dyeings would have been improved by a longer dyeing time. An increase in temperature would also have improved the dyeing, as discussed earlier and as illustrated in Figures 11 and 12. Again, this is evidence that equal or better penetration can be obtained by the high-temperature dyeing technique in a matter of minutes than is obtained in a matter of hours under the customary dyeing conditions.

In the application of chrome dyes by the top chrome method to nylon and wool, the use of the high-temperature technique is of value in obtaining rapid dyeing of the dye before chromation. So far, the evidence shows no marked advantage in carrying out the chromation at high temperatures.

The penetration of dye by the high-temperature technique, even into thick fabrics, is illustrated by a dyeing of a wool hat felt which was about ⅛ inch in thickness. Complete penetration was obtained when this material was dyed in a 3% solution of Calcocid Blue SAPG for 30 seconds at 270°F. A cross section at low magnification is shown in Figure 31, in which the extent of penetration is clearly evident. Commercial applications at present require long periods at the boil to obtain equivalent penetration.

It is evident from the experiments discussed above on the dyeing of cotton and rayon that the high-

temperature technique does not offer the same marked increase of development of color value over the normal procedures as in the case of the nitrogenous fibers. The rate of diffusion of most dyes into cellulosic fibers is quite rapid at the temperatures commonly used today. For this reason, continuous dyeing processes for cellulosic materials have been in existence for some time. Experiments have indicated that the high-temperature technique may give better penetration. In the present practice of dyeing mixed fibers—for example, cotton and wool unions—it is often difficult to obtain good unions because of the slower dyeing of the wool in comparison to the more rapid dyeing of the cotton. By the high-temperature technique, the rate of dyeing of the wool can be increased until it is similar to that of the cotton. For example, cotton and wool dyed for 20 seconds in 0.5% solution of Calcodur Orange GL showed the cotton to be dyed heavier than the wool at 230°F and 250°F, but at 270°F the wool was dyed heavier than the cotton. The color strength of the cotton dyeing did not vary to any marked extent as the temperature increased, but the color strength of the wool definitely increased with increase in temperature. The best union, therefore, would be obtained at some temperature between 250°F and 270°F when dyeing with this color. As another example, Calcomine Green BY Conc. showed similar color strength on cotton at the three temperatures, but the color strength of the wool did not equal that of the cotton until 270°F was used. Thus, the high-temperature dyeing technique makes possible better unions by a faster and simpler procedure.

The above examples are only a few of the many experiments which have been done using different types of dyes and variations in dyeing techniques at elevated temperatures. As in normal dyeing, there is considerable variation in the action of various dyes in the same and in different classifications. Some penetrate better than others, but in all cases the high temperature increases the rate of dyeing and diffusion into the fiber over that obtained at the present normal dyeing temperatures. In addition to the fibers mentioned above, successful dyeings have been made by the high-temperature method on immunized cotton, goat hair, cashmere, rabbit fur, linen, alpaca, and mohair. Also synthetic fibers such as the copolymer of vinyl chloride and vinylidine chloride and acrylonitrile polymer have been dyed. Others, such as the copolymer of vinyl chloride and vinyl acetate (Vinyon), were not successfully dyed because they fused at the high temperature.

Summary

Methods for dyeing textile fibers at elevated temperature in the range of 200°F to 300°F have been developed. The evidence presented shows that the high-temperature dyeing methods produce dyeings whose fastness, tensile strength, and other physical characteristics are equal or superior to those obtained by the normal dyeing procedures. This paper reports some of the factors affecting high-temperature dyeing. For example, the rate of dyeing is increased by an increase in the temperature. The time of dyeing is decreased by an increase in temperature. The pH of the dye bath must be controlled to obtain satisfactory dyeings. The amount of thermal decomposition of a large number of dyes was determined and those dyes most suitable for high-temperature dyeing were found. The high-temperature technique increases the speed of dyeing to such an extent that dyeing which normally requires hours can be produced in a matter of seconds. This increased speed of dyeing makes it practical to dye continuously at a rapid rate many fibers which previously have not been dyed by a rapid continuous process.

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(Manuscript received August 24, 1948.)

T. R. J. Covered by Index Services

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A Laboratory Study of Padding

Part I—Cotton

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Introduction

There has been considerable emphasis in the textile industry during the past few years on high-speed continuous dyeing processes. Most continuous dyeing methods for piece goods make use of padders or squeeze rolls at one or more stages in the processing of the fabric. Even though these operations are quite important in textile processing, there has been a great deficiency in fundamental knowledge concerning the principles of padding and liquid removal by squeeze rolls.

Padding is essentially a process which consists of saturating a fabric with a liquid (solution or dispersion) and then eliminating the excess by a passage between squeeze rolls. This usually produces a well-penetrated, evenly treated piece of goods. Padding is employed in both finishing and dyeing as a means of adding to the cloth a measured quantity of chemicals or dyes [8]. Owing to the short time of contact of the material with the dye solution, padding as a self-contained dyeing process is used only for dyeing light shades, or when maximum fastness must be sacrificed to rapid, low-cost production [1]. Ordinarily, in vat dyeing the padder is used as a means of rapidly obtaining a uniform deposition of the dye over the entire surface of the cloth; the diffusion or dyeing step then takes place in another machine.

The amount of dye deposited on a particular fabric during the padding operation depends primarily upon (1) the concentration of dye in the pad bath, (2) the percent pick-up, and (3) the ratio of solute (dye) to solvent (water) picked up—that is, the amount of preferential sorption of either solvent or solute. The first of these three variables is easily controlled in the preparation of the padding solution. The present study covers the factors which control the last two variables—that is, pick-up and preferential sorption.

The construction of certain fabrics together with the nature of various dyeing processes often makes it desirable to be able to increase the pick-up. When dyeing very deep shades, an increase in the pick-up would permit the use of a more dilute padding solution. This may be quite important when working near the limit of the solubility of a dye. An increase in pick-up may be desirable also when applying certain finishing compounds in order to get a greater concentration of finishing material on the fabric.

On the other hand, it is often desirable to decrease the pick-up. This is especially true when padding or squeezing is to be followed by drying. A lower pick-up results in lower drying costs because the cost for water removal by squeezing is far less than by heat. In some continuous dyeing ranges, no provision has been made for drying the cloth between the padder and the developing bath. Under these circumstances, the less moisture the cloth retains as it leaves the padder the greater and faster is the pick-up of developing chemicals. In some cases, a decrease in the pick-up may decrease the migration of non-substantive dyes before drying.

Most of the results in this investigation are reported as "percent pick-up." *The percent pick-up is defined as the weight of the pick-up multiplied by 100 divided by the weight of the cloth before padding.*

The prevention of preferential sorption of either the dye or the dye solvent during padding is desirable. If exhaustion of the dye occurs in the pad box, the padding solution becomes progressively weaker and the shade of the padded cloth becomes progressively lighter. When the dye solvent is preferentially sorbed, the shade of the padded cloth

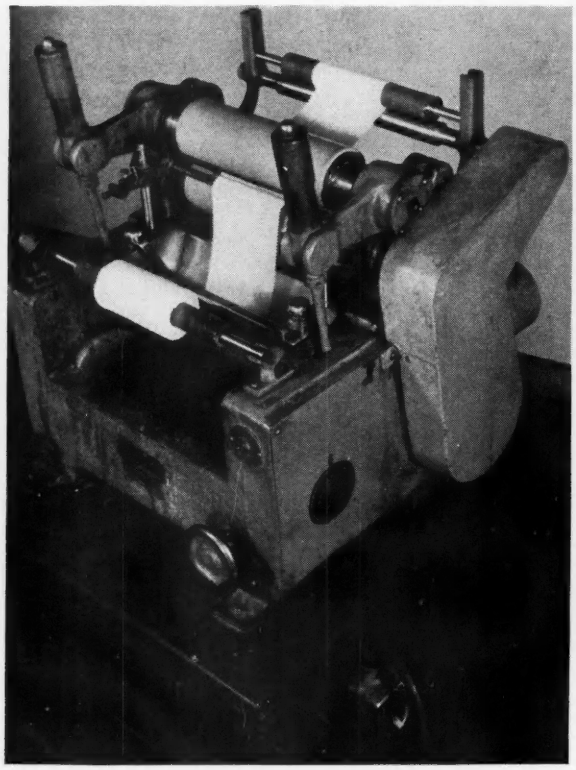

FIG. 1. *Butterworth Laboratory two-roll padder.*

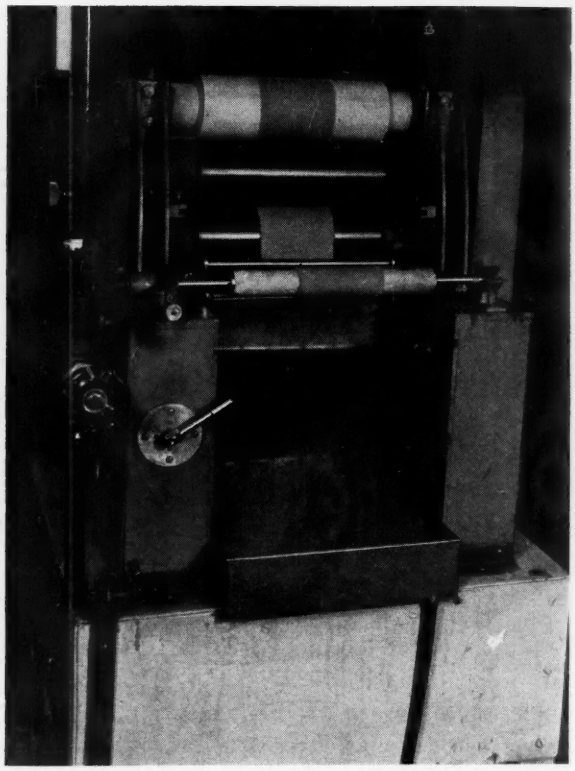

FIG. 2. *Butterworth Laboratory three-roll padder.*

becomes progressively heavier. This makes it necessary to prepare feed baths having a different proportion of dye from the starting bath, if shading from start to finish is to be avoided. Ideal padding would be a purely mechanical operation with both solvent and solute (dissolved dye or dispersed pigment) taken up in amounts proportional to their concentration in the padding solution.

In order to express preferential sorption mathematically, a new term, "dye pick-up factor" (abbreviated D.P.F.), is proposed. *The D.P.F. is defined as the ratio obtained by dividing the amount of dye actually on the cloth, as determined by analysis, by the amount of dye that should be on the cloth, as calculated from the pick-up, and then multiplying this fraction by 100.* Thus, a D.P.F. greater than 100 shows that dye was exhausted from the pad box. A D.P.F. less than 100 shows that water was preferentially sorbed. In ideal padding the dye and water are picked up at the same rate, giving a D.P.F. of 100.

This study of padding has been approached by changing the conditions of padding in such a way that in each set of experiments all variables were held constant except one. This single variable was changed over a wide range and the effect of these changes upon the percent pick-up and the D.P.F. was determined. The variables in padding fall into three classes: those in which (1) the cloth is varied, (2) the dye bath is varied, and (3) the padder is varied.

The cloth can be varied in its chemical constitution by using in its construction protein fibers, cellulosic fibers, or various synthetic fibers. The physical constitution of the cloth can be varied by weave, size, and twist of yarn, etc. The cloth may be changed by various kinds of treatments before padding, such as desizing, scouring, bleaching, mercerizing, etc. The cloth may also be treated with wetting agents and dried before padding.

When padding with a soluble dye, both the kind and the concentration of dye as well as the solvent can be varied. When using pigment dispersions, for example, in vat pigment padding, both the pigment and the vehicle can be varied. In addition, the pH, temperature, and viscosity of the bath can be changed. Various assistants, such as wetting agents, can be added to the padding bath.

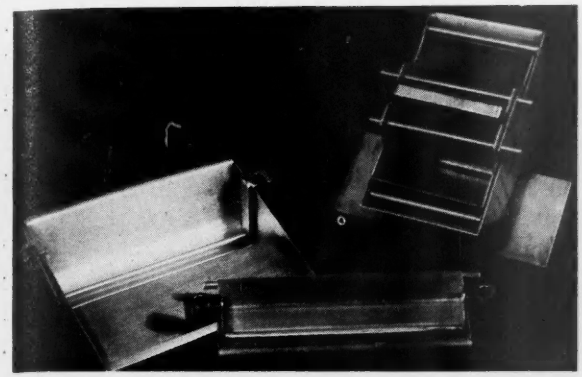

FIG. 3. *Interchangeable pad boxes for two-roll padder.*

The padder may be varied with respect to the speed, pressure, number of dips and nips, and time of immersion. The size, number, and composition of the squeeze rolls are also variable.

The above paragraphs list some of the variables in padding. An attempt has been made to study the relation of some of these variables to the pick-up and the D.P.F. The published literature on the fundamental aspects of padding in the textile industry is quite limited. The *Kenwood Bulletin Book* [2] is an excellent general treatise, written for the paper industry, on water removal by squeeze rolls. Some of the theory presented in this book has been applied to the interpretation of the experimental data obtained in this investigation of textile padding. However, there is still much in the way of fundamental knowledge of padding that is needed urgently in the industry. Furthermore, these data, obtained in the laboratory, need to be checked on plant equipment under commercial dyeing conditions.

Apparatus and Equipment

All of the experimental work was carried out on a Butterworth two-roll padder equipped with a General Electric Thy-mo-trol speed control (shown in Figure 1). The speed of this padder could be varied from 1 yard per minute to 44 yards per minute. The padder was run at 20 yards per minute except where otherwise noted. The padder had three interchangeable pad boxes of 150-ml., 500-ml., and 1,500-ml. capacity (see Figure 3). Several rolls (see Figure 4) were available for the padder which varied widely in hardness, as shown in Table I. Two soft rubber rolls were used in all experiments except where otherwise noted. Pressure was applied to the squeeze rolls of this padder by compressing springs which are at the end of lever arms connected to each end of the roll. This was done in one operation by turning a hand wheel which loads both ends of the rolls equally.

TABLE I. ROLLS USED FOR THE PADDER

Roll	Hardness of roll		
	Shore Durometer	Adams Densimeter	Shore Elastometer
Top roll (soft rubber)	71	34	38
Top roll (hard rubber)	96	12	14
Bottom roll (soft rubber)	73	32	74
Bottom roll (hard rubber)	95	11	17
Bottom roll (steel)	102	2	—

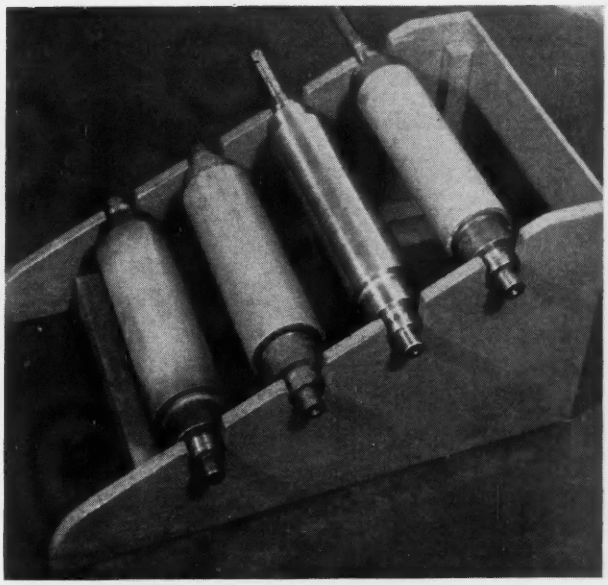

FIG. 4. *Interchangeable rolls of varying hardness for two-roll padder.*

Quantitative Determination of Dye on Cloth

Extraction Procedure for Direct Dyes

The padded cloth to be extracted was conditioned for 24 hours at 70°F and 65% relative humidity. A quantitatively weighed sample of 0.3 g. to 0.4 g. was cut from the conditioned padding. The dye was removed from this weighed sample by refluxing for 10 minutes with 50 ml. of a 1:1 pyridine-water solution. The solvent was poured off and the extraction was repeated with fresh pyridine-water solution. The two extractions were combined, diluted with pyridine-water solution in a volumetric flask to

a suitable concentration, and the concentration was measured by a spectrophotometer or colorimeter.

Extraction Procedure for Vat Dyes *

(*a*) *Pigment Paddings.* The padded fabric to be extracted was conditioned for 24 hours at 70°F and 65% relative humidity. A sample 0.3 g.–0.6 g. was cut from the conditioned sample and weighed. The dye was removed by allowing the sample to simmer for 15 to 20 minutes in about 30 ml. of dry pyridine. The process of simmering was repeated with fresh pyridine until all the dye was removed; usually twice was sufficient. The combined extracts were diluted to a suitable concentration quantitatively with pyridine, and the concentration of dye in the solution was determined by spectrophotometric measurement.

(*b*) *Leuco and Soluble Vat Paddings.* The padding to be extracted was conditioned for 24 hours at 70°F and 65% relative humidity. A sample weighing 0.3 g.–0.6 g. was cut from the conditioned padding. Five milliliters of water and 2–3 drops of $5N \cdot H_2SO_4$ were added to the sample, which was then heated by steam until almost dry. The dye was then removed by allowing the sample to simmer for 15 to 20 minutes in about 30 ml. of pyridine. The process was repeated with fresh pyridine, combining the extracts, until the color was removed from the sample. Two extractions were usually sufficient. The combined extracts were diluted quantitatively with pyridine to a suitable concentration and the concentration of dye in the solution was determined by spectrophotometric measurement.

Cloths

Most of the experiments were done using either $80 \times 80 - 4.00$ bleached, unmercerized cotton percale or a $112 \times 46 - 3.50$ unbleached and unmercerized cotton poplin. The poplin was used in most of the experiments because the percale wet out so readily under even the poorest conditions that it was not a good fabric to use to study variations in pick-up.

Padding Procedures

A piece of cloth about 4 inches wide and 24 inches long was weighed on an analytical balance. Leaders were sewed lightly, by hand, to each end of the cloth and one of these was threaded through the padder. The padding solution was heated to a temperature slightly higher than the required temperature and placed in the pad box. When the temperature of the solution dropped to the desired temperature, the padding was made. The leaders were removed carefully by cutting the sewing threads and the padded cloth was weighed at once. The padding was dried and placed in the conditioning room at 70°F and 65% relative humidity to await analysis.

The pad liquor, except where otherwise noted, was as follows:

A. Direct dyes
   5.0 g. dye
   4.0 ml. Aerosol OT—10% clear
   Water to make 500 ml.

B. Vat dyes
   1. Pigment padding
      5.0 g. dye
      Water to make 500 ml.
      Stirred 5 minutes immediately before using.
   2. Padding of vat leucos
      5.0 g. dye
      3.0 g. NaOH
      4.0 ml. Igepon T Gel (1% solution)
      Water to make 500 ml.
      Heated to 140°F, 5.0 g. $Na_2S_2O_4$ added, and reduced at 140°F for 5 minutes.
   3. Soluble vats
      2.5 g. dye
      4.0 ml. Aerosol OT—10% clear.
      Water to make 500 ml.

Precision of Padding Data

There is a limit to the exactness with which any observation or measurement can be made. When a quantity is measured with the greatest exactness of which the instruments and the method used are capable and with the greatest skill that the operator

TABLE II. PRECISION OF % PICK-UP AND D.P.F. MEASUREMENTS

Padding	Standard deviation	
	% Pick-up measurements	D.P.F. measurements
Direct dyes	1.7	4.6
Leuco vats	1.8	3.0
Pigment vats	1.0	3.0
Soluble vats	2.0	3.0

* Details to appear in a future publication from these laboratories.

TABLE III. EFFECT OF CLOTH CONSTRUCTION ON PADDING

Type of cloth	Calcomine Navy R		Calcosol Navy Blue paste				Calco Soluble Vat Pink FF	
			Pigment padding		Leuco padding			
	% Pick-up	D.P.F.	% Pick-up	D.P.F.	% Pick-up	D.P.F.	% Pick-up	D.P.F.
Percale	73	102	68	91	67	169	77	87
Poplin	18	109	19	88	22	173	19	100
Duck	6	57	4	94	8	308	6	92

can exercise, it is found that the results of successive measurements still will differ by a finite amount. The reproducibility of such a series of measurements is called the "precision." The precision of a series of measurements made under the same conditions may be expressed as the standard deviation. If the number of measurements is small, the standard deviation cannot be accurately determined by any formula, but a sufficiently good approximation of the standard deviation can be obtained from the following formula:

$$\text{Standard deviation} = 5/4 \frac{\text{Sum of deviations of each measurement from average of all measurements}}{\text{Number of measurements} - 0.8}$$

Using this formula, the standard deviations for both percent pick-up and dye pick-up factor were determined and are shown in Table II.

Results and Discussion

This section, which contains the experimental results and a discussion of these results, is divided into three parts: (1) variations in the cloth, (2) variations in the dye bath, and (3) variations in the padder.

1. The Cloth

It is well known that the percent pick-up and penetration varies when fabrics of different constructions and weights are padded. For example, a piece of duck is much harder to penetrate than a piece of percale. One reason for this difference is the speed of wetting. A lightweight fabric wets out very rapidly, whereas a heavy fabric may require a very long time to wet out completely. A second factor is the porosity of the cloth. As the liquid is released farther back in the nip, it is necessary for the liquid to filter back through the fabric in the opposite direction to the fabric's motion. Table III gives the variation in percent pick-up and D.P.F. obtained when padding three different types of cloth with the same dye solutions. The data show that a smaller pick-up is obtained from heavy fabrics made of tightly twisted yarn than from lightweight fabrics.

Since one object of padding is the uniform distribution of dye over the cloth, it is important that the cloth be in a highly absorbent condition before padding. This absorbent condition of the cloth can often be obtained by an efficient scouring; this is especially true in the case of lightweight, low-count fabrics. When heavy, hard-to-penetrate fabrics are padded, a pretreatment with certain wetting agents will increase the percent pick-up with little effect upon the D.P.F. Table IV shows the change in percent pick-up and D.P.F. caused by pretreating various types of cloth with 2 oz. per gal. Aerosol OT (10%) at 70°C (158°F) and drying before padding with dye. It can be seen that little difference in pick-up results from pretreating the light-

TABLE IV. EFFECT OF PRETREATING WITH AEROSOL OT

Cloth	Calcomine Navy R		Calcosol Navy Blue paste				Calco Soluble Vat Pink FF	
			Pigment padding		Leuco padding			
	% Pick-up	D.P.F.	% Pick-up	D.P.F.	% Pick-up	D.P.F.	% Pick-up	D.P.F.
Percale								
Pretreated	72	101	71	86	67	164	74	93
Untreated	73	102	68	91	67	169	77	87
Poplin								
Pretreated	52	97	56	89	50	133	52	92
Untreated	18	109	19	88	22	173	19	100
Duck								
Pretreated	11	117	14	72	10	270	14	99
Untreated	6	57	4	94	8	308	6	92

TABLE V. EFFECT OF DYE CONCENTRATION UPON % PICK-UP AND D.P.F.

Concentration of dye (g./100 ml.)	Calcodur Pink 2BL		Calcosol Navy Blue paste				Calco Soluble Vat Pink FF	
			Pigment padding		Leuco padding			
	% Pick-up*	D.P.F.	% Pick-up*	D.P.F.	% Pick-up*	D.P.F.	% Pick-up*	D.P.F.
0.25			67	97	70	240	33	130
0.50	27	124	68	94	68	208	34	120
1.0	29	107	67	95	68	175	34	109
2.0	30	100	68	96	66	149	36	70
4.0	30	84	69	89	72	113	30	55
8.0	31	82	68	86	75	89	31	47

* The differences in percent pick-up are due to the use of poplin when padding the two pink dyes and percale when padding the blue dyes.

weight percale because this fabric wets out completely without any pretreatment; however, a marked increase in pick-up was obtained by pretreating the heavier poplin and duck.

2. The Dye Bath

Just as in dyeing, padding varies with the class of dye used. This investigation included the padding of direct dyes, soluble vat dyes, leuco vat dyes, and vat pigment dispersions. In each of these classes, one or two dyes were used in all the tests as representative dyes of their class. It is recognized that each dye acts as an individual and is different from other dyes in the same class; however, in any test, differences between dyes in the same class will be one of degree and not one of kind.

The concentration of the dye in the pad liquor has little or no effect upon the percent pick-up regardless of the type of dye used. As the dye concentration in the pad liquor is increased, the D.P.F. decreases only slightly in the case of vat pigment dispersions, but decreases quite markedly in the case of direct dyes, soluble vats, and vat dyes padded in the leuco state. When padding with water-soluble dyes, the dye should be in complete solution if uniform paddings are desired. The effect of the dye concentration upon the percent pick-up and D.P.F. is shown in Table V.

TABLE VI. EFFECT OF pH OF DYE BATH

pH of dye bath	Calcomine Navy R		Calcosol Navy Blue paste Pigment padding	
	% Pick-up	D.P.F.	% Pick-up	D.P.F.
2	43	133	67	106
4	43	132	67	96
6	43	131	65	96
8	44	126	66	98
10	43	127	66	99
12	43	128	65	92

A change in the pH of the dye bath appears to have little effect upon the percent pick-up or D.P.F. in the padding of cotton fabrics with direct dyes and vat pigment dispersions, as is illustrated in Table VI.

When padding leuco vats, a variation in the concentration of caustic soda and sodium hydrosulfite appears to have no effect on either the percent pick-up or the D.P.F. as long as there is enough caustic soda and sodium hydrosulfite present to keep the dye in a reduced state. Table VII shows that no change was obtained in either the percent pick-up or the D.P.F. by varying the caustic soda and sodium hydrosulfite concentration when padding a 1% solution of Calcosol Navy Blue leuco at 140°F.

It appears that the variation in pick-up with the temperature of the pad bath is affected more by the type of fabric used than by the type of dye used. When padding lightweight, easy-to-wet fabrics, the pick-up decreases as the temperature of the pad liquor increases. This tendency for the pick-up to decrease as the temperature of the pad bath increases is counteracted, when padding heavy, hard-to-wet fabrics, by the increased rate of wetting, so that, in some cases, the pick-up actually increases as

TABLE VII. VARYING NaOH AND $Na_2S_2O_4$ CONCENTRATIONS

Concentration of dye (g./100 ml.)	Concentration NaOH (g./100 ml.)	Concentration $Na_2S_2O_4$ (g./100 ml.)	% Pick-up	D.P.F.
1	1	1	71	163
1	3	3	71	174
1	5	5	71	170
1	10	10	72	168
1	3	1	71	160
1	3	5	71	181
1	3	10	71	176
1	1	3	70	170
1	5	3	71	169
1	10	3	72	165

TABLE VIII. EFFECT OF TEMPERATURE AND FABRIC UPON PADDING

Temperature of padding bath	Calcosol Navy Blue paste							
	Pigment padding				Leuco padding			
	Percale		Poplin		Percale		Poplin	
	% Pick-up	D.P.F.	% Pick-up	D.P.F.	% Pick-up	D.P.F.	% Pick-up	D.P.F.
40°C	67	94	31	83	73	153	32	113
60°C	62	103	31	85	69	174	37	126
80°C	59	110	30	92	66	211	31	131

the temperature is increased. This is shown in Table VIII.

The D.P.F. always seems to increase as the temperature of the padding bath is increased. The amount of the increase depends upon the dye. This increase of D.P.F. with temperature is due probably to an increase in the rate of dyeing usually associated with a temperature rise. Table IX shows the change in pick-up and increase in D.P.F. that were obtained when the temperature of the pad bath was increased.

The addition of wetting agents to the pad solution will increase the percent pick-up when padding fabrics which are difficult to wet out or penetrate. When using lightweight fabrics, the addition of a wetting agent has little or no effect on the percent pick-up since these fabrics wet out rapidly and completely without the aid of such assistants. The addition of a wetting agent usually has no effect upon the D.P.F.; however, in the case of vat leucos, the dyeing rate is usually reduced by these agents and, as a result, the D.P.F. is decreased. The increased pick-up obtained on poplin by adding various amounts of Aerosol OT (10% clear) to the pad bath are shown in Table X.

An increase in the viscosity of the pad bath usually results in an increase in the percent pick-up. The D.P.F. does not appear to be affected uniformly by viscosity changes. Table XI shows the increased pick-up produced by a viscosity change when padding cotton poplin with dye baths whose viscosities have been varied by the addition of gum tragacanth.

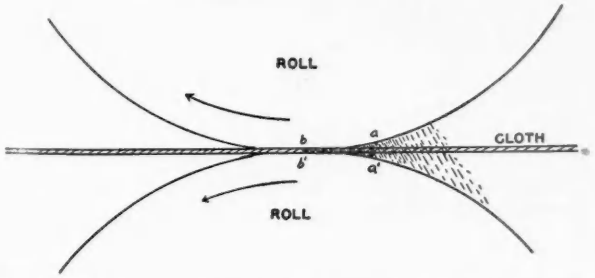

FIG. 5. *Diagrammatic sketch showing conditions at nip during padding.*

3. The Padder

There are several factors which determine the amount of liquid removed from or retained by the cloth during a passage through the squeeze rolls of a padder. Some of these factors are:

1. The condition of the cloth or the degree to which it is wet out.
2. The construction of the cloth or resistance of the fabric to the flow of liquid.
3. The velocity of the cloth through the rolls.
4. The pressure between the squeeze rolls, which is controlled by the force applied, the hardness of the rolls, the size of the rolls, and the thickness of the rubber covering.

As cloth passes between the squeeze rolls of a padder, the dye solution begins to be squeezed out at points *a, a'*, as shown in Figure 5. From this point to *b, b'*, the pressure becomes progressively greater, reaching a maximum at *b, b'*. As the cloth approaches *b, b'*, there are several forces working on the cloth-dye solution system simultaneously. There is resistance to dye solution passing through the nip due to pressure between the rolls. There is a force in the opposite direction which is the tendency of the liquid to carry on through the nip in the direction of the running cloth; this force varies with the speed of the padder. There is also the tendency for the revolving rolls to carry the dye solution into the nip. As this dye solution is released closer and closer to the center of the nip, there is more and more necessity for the solution to have to filter back through the fabric in a direction contrary to the fabric's motion. Thus, the resistance of the fabric to the flow of liquid is another force acting on the system. Equilibrium in this system will depend upon the balance between these forces.

The percent pick-up decreases as the force per unit area between the squeeze rolls increases. Our investigation has shown that the effective pressure (lb. per sq. in.) between the pad rolls depends upon three things: (1) the force applied, (2) the hardness

TABLE IX. EFFECT OF TEMPERATURE UPON PADDING

Temperature of dye bath	Calcomine Navy R		Calcosol Navy Blue paste Pigment padding		Calcosol Navy Blue paste Leuco padding		Calco Soluble Vat Pink FF	
	% Pick-up*	D.P.F.	% Pick-up*	D.P.F.	% Pick-up*	D.P.F.	% Pick-up*	D.P.F.
30°C	33	101	71	93	77	140	34	89
40°C	33	102	67	94	72	153	35	85
50°C	33	108	64	98	73	145	35	89
60°C	34	108	62	103	69	174	36	94
70°C	35	107	62	103	68	193	38	93
80°C	40	111	59	110	66	211	39	95

* The differences in percent pick-up are due to the use of poplin when padding Calcomine Navy R and Calco Soluble Vat Pink FF. Percale was used in padding the Calcosol Navy Blue paste.

TABLE X. USE OF AEROSOL OT IN PADDING

Aerosol OT (10% clear) (g./l.)	Calcomine Navy R		Calcosol Navy Blue paste Pigment padding		Calcosol Navy Blue paste Leuco padding		Calco Soluble Vat Pink FF	
	% Pick-up	D.P.F.	% Pick-up	D.P.F.	% Pick-up	D.P.F.	% Pick-up	D.P.F.
0	22	101	19	93	22	184	22	92
5	29	100	26	98	23	179	35	86
10	36	96	32	97	28	153	37	86
20	42	97	38	96	32	144	44	89
40	53	99	50	95	41	127	54	90
80	55	102	58	99	52	114	57	87

TABLE XI. EFFECT OF VISCOSITY UPON PADDING

Viscosity of pad bath (c.p.s.)	Calcodur Pink 2BL		Calcosol Navy Blue paste Pigment padding		Calco Soluble Vat Pink FF	
	% Pick-up	D.P.F.	% Pick-up	D.P.F.	% Pick-up	D.P.F.
10	35	101	31	98	37	84
20	38	98	33	96	41	95
30			35	92	42	88
50	41	99	37	108	44	94
100	44	96	39	107	44	83
250	46	93	41	98	46	88

TABLE XII. EFFECT OF PRESSURE AND ROLL COMPOSITION ON PADDING

Composition of pad rolls	Pressure dial setting on padder 1			2			3			4			5		
	Pressure (lb./sq.in.)	% Pick-up	D.P.F.	Pressure (lb./sq.in.)	% Pick-up	D.P.F.	Pressure (lb./sq.in.)	% Pick-up	D.P.F.	Pressure (lb./sq.in)	% Pick-up	D.P.F.	Pressure (lb./sq.in.)	% Pick-up	D.P.F.
Hard rubber against steel	28	56	132	111	49	126	155	46	131	203	45	125	206	42	133
Hard rubber against hard rubber	33	64	98	99	49	102	154	45	109	187	44	108	188	44	109
Soft rubber against steel	25	72	102	83	61	107	122	57	110	151	52	114	154	52	101
Soft rubber against hard rubber	20	73	92	71	61	95	100	58	98	125	52	105	129	52	109
Soft rubber against soft rubber	27	66	94	55	55	102	86	54	103	102	49	106	106	51	97

TABLE XIII. DETERMINATION OF PRESSURE BETWEEN PAD ROLLS

Pressure dial setting on pad	*D* Distance spring is compressed (in.)	*M* Mechanical advantage	*K* Force constant	Area of nip (sq. in.)	Pressure between rolls (lb./sq. in.)
1	0.17	2.83	88	3.13	27
2	0.59	2.83	88	5.32	55
3	1.02	2.83	88	5.94	86
4	1.34	2.83	88	6.56	102
5	1.46	2.83	88	6.88	106

TABLE XIV. EFFECT OF SPEED UPON PADDING

Speed of padder (yd./min.)	Time of immersion (sec.)	Calcomine Navy R		Calcosol Navy Blue paste				Calco Soluble Vat Pink FF	
				Pigment padding		Leuco padding			
		% Pick-up	D.P.F.	% Pick-up	D.P.F.	% Pick-up	D.P.F.	% Pick-up	D.P.F.
10	0.7	27	113	27	97	19	156	29	100
20	0.7	30	113	29	94	24	143	32	88
40	0.7	33	111	32	82	27	149	36	82

TABLE XV. EFFECT OF IMMERSION TIME UPON PADDING

Speed of padder (yd./min.)	Time of immersion (sec.)	Calcomine Navy R		Calcosol Navy Blue paste				Calco Soluble Vat Pink FF	
				Pigment padding		Leuco padding			
		% Pick-up	D.P.F.	% Pick-up	D.P.F.	% Pick-up	D.P.F.	% Pick-up	D.P.F.
3	1.82	32	105					35	84
3	4.75	39	99					41	81
3	9.60	40	104					45	81
28	0.21	25	109	20	98	23	114	28	99
28	0.54	32	100	20	94	22	156	32	93
28	1.09	35	101	22	77	25	154	37	89
45	0.13	26	105	20	105	23	108	27	96
45	0.34	31	103	21	88	27	126	33	91
45	0.69	35	100	22	82	27	156	36	88

TABLE XVI. EFFECT OF DIPS AND NIPS UPON PADDING

Number of dips and nips	Calcomine Navy R		Calcosol Navy Blue paste				Calco Soluble Vat Pink FF	
			Pigment padding		Leuco padding			
	% Pick-up	D.P.F.	% Pick-up	D.P.F.	% Pick-up	D.P.F.	% Pick-up	D.P.F.
1	33	102	32	80	20	154	35	89
2	49	96	46	83	35	156	48	80
3	54	104	55	81	47	150	51	74
4			56	83	53	152	52	82

of the rolls, and (3) the size of the rolls. A fourth variable affecting the pressure—namely, the thickness of the rubber covering—has been reported in the literature [7].

There is a difference between the force applied to the rolls and the effective pressure. The same total force could be applied to two sets of rolls, but if one set of rolls were covered with softer rubber, the soft rolls would flatten more, spreading the force over a larger area, and thus give a smaller effective force per unit area of nip. The hardness of the rolls, then, affects the pressure, greater liquid removal for a given pound per linear inch being obtained as one approaches the straight-line contacts produced by extremely hard rolls. Thus, the dryness of the cloth varies directly with the sum of the hardnesses of the two rolls [3]. The nip width increases directly with roll plasticity [4]. Figure 6, taken from the *Kenwood Bulletin Book,* shows graphically the relationship between the width of the nip and the pressure, using rubber-covered rolls of varying hardness. Theoretically, when cloth of ap-

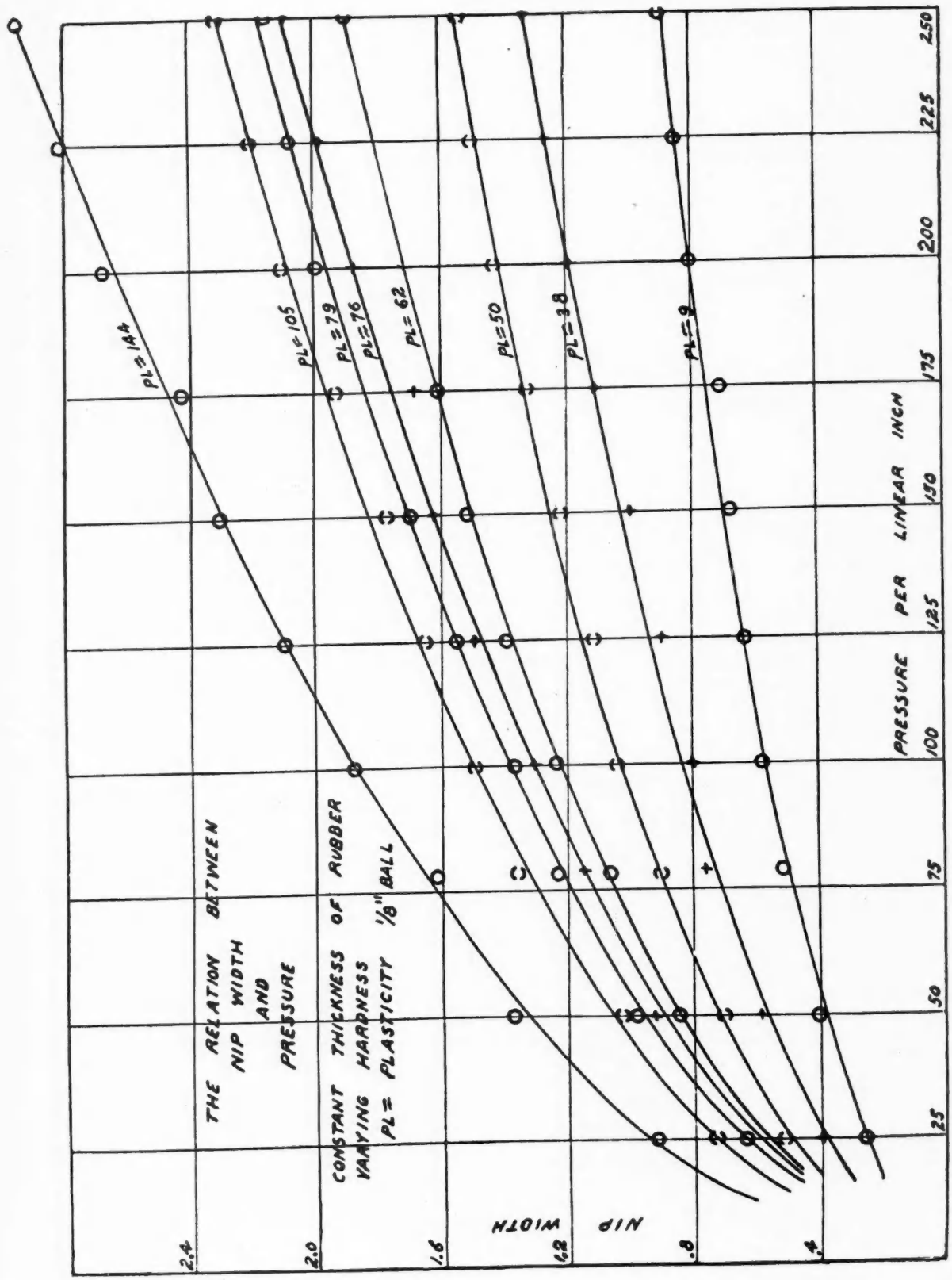

FIG. 6. *Relationship between nip width and pressure using rolls of different hardness.* (Courtesy of F. C. Huyck and Sons.)

preciable thickness is padded, two rolls of unequal hardness will give different pressures on the back and face of the cloth. For example, suppose the top roll is harder than the bottom roll; in contact with the cloth the hard top roll gives the smaller contact zone against the cloth, which results in a greater pressure and a more concentrated squeezing. Table XII shows the results obtained by padding 2.50 cotton drill with a 1% solution of Calcomine Navy R using different pad rolls (and shows the decrease in pick-up obtained as the pressure (lb. per sq. in.) is increased).

The force per unit area (lb. per sq. in. pressure) was calculated by substitution in the following formula:

$$\text{Pressure at nip (lb. per sq. in.)} = \frac{2\ KDM}{A},$$

where K is the force constant of the springs used to apply force to the laboratory padder expressed in lb. per in., D is the distance in inches that the spring is compressed, M is the mechanical advantage gained through the lever arms, and A is the area of the nip in square inches. The right side of the equation is multiplied by 2 since there are two springs.

The force constant of each spring, K, was determined by finding the weight or force required to compress the spring several measured distances, and, from these data, K, the force required to compress the spring 1 inch, was calculated. The force constants of the two springs were so nearly alike that the two values were averaged for use in the above equation. For the laboratory padder used in this work, K was 88 lb. per in.

The area of the nip was determined by measuring the area of an imprint of the nip contact zone of the rolls at different applied pressures. The imprints were made by putting sheets of carbon paper (Hecto) between damp sheets of white paper and then placing this sandwich between the rolls. On applying pressure, a print of the nip contact zone is made on the white paper. Table XIII shows the data used in calculating the pressure between the rolls of the laboratory padder when two soft rubber rolls were used.

It has been pointed out in the previous paragraph that the pick-up is governed by the effective pressure (lb. per sq. in. of nip area) rather than the total force applied to the rolls. A fundamental law has been found [4] which states that the nip width increases directly as the square root of the force per linear inch. This law is based on rolls having very thick rubber coverings and does not necessarily hold true for rolls having a thinner covering. Although the area of the nip increases as the total force applied to the rolls increases, the increase in nip area is usually not in proportion to the increase in pressure. Thus, in general, an increase in force gives a greater concentration of force per square inch of nip area, resulting in a decrease in the pick-up. However, it has been found that in some cases an increase in pressure does not give a decrease in pick-up [5]. The pick-up depends not only upon the pressure but also upon the resistance of the fabric to the flow of liquid. The liquid has to filter back through the fabric in the area of the nip contact; thus, when additional pressure pinches off the channel, making a still wider nip, the liquid is prevented from flowing away, owing to the resistance of the fabric. This happens most often when soft rolls and very wide nip areas are used. Thus, for every fabric and combination of rolls there is probably an optimum pressure beyond which no additional liquid removal can be obtained.

The size of the pad rolls has an important bearing upon the pick-up. The sizes of the rolls are often governed by material and mechanical considerations, but when considered purely from the viewpoint of pick-up, the larger the roll the greater the width of the nip. This means that, using the same total applied force, a large roll will give a lower pressure (lb. per sq. in.) and a longer path for the liquid to filter through than a small roll. The nip widths are directly proportional to the diameter of the rolls if the two rolls are of the same diameter. If a combination of two rolls of different diameter is used, the nip width will lie between the values for the separate diameters [6].

The thickness of the rubber covering on the pad roll is an exceedingly important factor, and yet it has received very little attention. The Kenwood Laboratories have carried out much work in studying the relation between the thickness of the rubber covering on paper press rolls and water removal [7]. The results of their tests, shown in Figure 7, show that the nip width varies directly with the thickness of the rubber on the roll. Thus, as far as pick-up is concerned, increasing the thickness of the rubber covering on the pad roll has the same effect as increasing the softness of the rubber—that is, increasing the pick-up.

FIG. 6. *Relationship between nip width and pressure using rolls of different hardness.* (Courtesy of F. C. Huyck and Sons.)

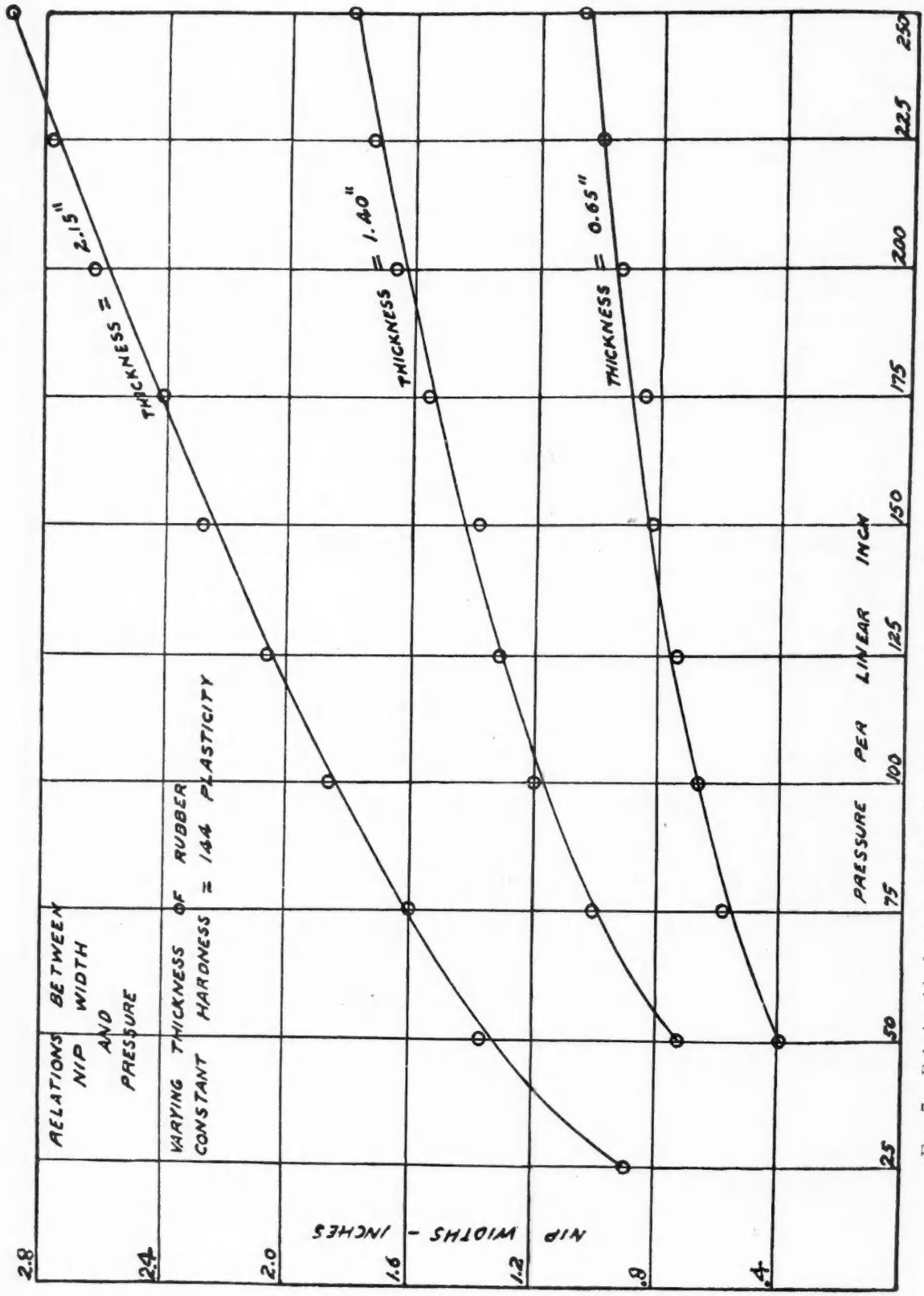

FIG. 7. *Relationship between nip width and pressure using varying thicknesses of rubber covering on the rolls.* (Courtesy of F. C. Huyck and Sons.)

The pick-up is also affected by the speed of the padder. Using the same pad box, the time of immersion is decreased as the speed of padding is increased. If the size of the pad box is varied, it is possible to vary the speed of padding and at the same time maintain a constant time of immersion. Experiments show that the pick-up or tendency of the liquid to carry on through the nip in the direction of the moving fabric is usually increased by an increase in the speed of padding. The increase in pick-up with speed is probably due to two things: (1) the force with which the liquid moves forward owing to its own velocity, and (2) the time of contact at the nip. The time of nip contact is important because of the resistance to any change in conditions due to the inherent inertia of the cloth-liquid system and the lag between application of the forces and their effects. In general, the D.P.F. remains constant or decreases slightly as the speed of padding is increased. Table XIV shows how the pick-up increases and the D.P.F. varies as the speed of padding varies from 10 to 40 yards per minute when padding cotton poplin with various dyes.

Both the pick-up and D.P.F. are affected by the time of immersion. The pick-up increases with longer immersion times because the cloth has more time to wet out. This is especially true when padding fabrics which are slow or difficult to wet. When padding lightweight fabrics, such as percales, an increase in the time of immersion has little effect upon the pick-up since these lightweight fabrics are wet out almost instantaneously. Other fabrics, such as heavy ducks, may have to be soaked for a much longer period to become completely wet. The change in D.P.F. with immersion time seems to depend somewhat upon the dyeing rate of the color used. When using dyes which have very fast rates of exhaustion, a larger D.P.F. is obtained as the time of immersion is increased. In the case of pigments, which have little affinity for the fiber, a smaller D.P.F. is obtained as the immersion time is increased, owing to the water molecules diffusing into the fibers and leaving the large dye molecules on the outside of the fiber to be washed off at the nip. Table XV shows the increase in percent pick-up and change in D.P.F. obtained when cotton poplin is padded using increasing immersion times.

When the number of dips and nips is increased, the time of immersion is also increased. Table XVI shows that the percent pick-up increases as the number of dips and nips is increased. The D.P.F. is apparently unaffected. These experiments were made on poplin. In other experiments using percale, the D.P.F. increased with increasing numbers of dips and nips.

Summary

Essentially, padding consists of two steps: (1) the wetting of the fabric with the dye solution and (2) its passage between squeeze rolls. During either or both of these steps, it is possible to have preferential sorption of either the solute (dye) or the solvent (water).

Some of the variables affecting the wetting of the fabric are the construction of the cloth, the condition of the cloth, the temperature of the pad bath, the addition of wetting agents to the dye bath, the time of immersion, the speed of the padder, and the number of dips and nips.

The amount of liquid expressed by the squeeze rolls is dependent not only upon the variables in the padder but also upon the wetness of the cloth. Some of the variables in the padder affecting the squeezing operation are the speed of the padder and the pressure (lb. per sq. in.) between the squeeze rolls; the pressure between the squeeze rolls depends upon the total force applied to the rolls, the hardness of the rolls, the size of the rolls, and the thickness of the rubber covering on the rolls.

It has been shown through the use of the "dye pick-up factor" (D.P.F.) that preferential sorption of either the dye or the vehicle may take place in padding. Factors which have been shown to affect the preferential sorption are the construction and constitution of the cloth, the dye, the temperature of the pad bath, the addition of assistants to the pad bath, the time of immersion, and the speed of the padder.

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(*Manuscript received by* Textile Research Journal, *Sept. 14, 1948.*)

Degree of Polymerization of Cellulose in Cotton Fibers

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THE PHYSICAL PROPERTIES of cotton are dependent on the fiber structure and shape. Their use value is also influenced by the appearance of the fibers and the products made from them. Fiber structure, as discussed here, consists of the size, shape, and position of the cellulose molecules and aggregates of molecules either as crystallites, fibrils, or regions of amorphous cellulose. A given cellulose molecule is composed of glucose anhydrides polymerized into a long chain the length of which is dependent on the degree of polymerization (designated "D.P.").

The length of the chain (D.P.) is known to vary in cotton fibers as used in commerce. These variables may be associated with the growth processes as the tissues are formed or later by degradation due to sunlight, microorganisms, and chemicals.

The cotton fiber develops in two or more distinct stages. The cell first grows in size, then in cell-wall thickness. As the cell is elongating it consists of a thin membrane 0.5 μ or less in thickness, designated "primary wall," which is filled with protoplasm. The secondary thickening is deposited on the inner surface of the primary wall [21]. (See Figure 1.)

The chemical constituents of the primary wall are different from those of the secondary thickening [32]. Furthermore, the long axes of the cellulose crystallites in the primary wall are arranged transverse to those in the central region of the secondary thickening [1, 7, 21]. Shrinkage studies indicate that the crystallites in the primary wall are relatively short and have a tendency to be randomly dispersed, whereas those in the secondary wall appear to be long, thread-like strands arranged more or less parallel with each other. It is further indicated that in both regions of the cell wall the crystalline strands are intermixed with amorphous cellulose [8]. There is no indication that the degree of polymerization of the cellulose increases after the cell wall is formed; therefore, such differences as are found are interpreted to be due to variations in the development of the fiber and to degradation which occurs during subsequent exposure.

Cotton fibers from different varieties [9] and locations of growth [22] also vary in physical properties; therefore it is necessary to distinguish between the effects of stage of development, or maturity of the fiber, and degradation which has taken place after the cellulose was formed. The data reported here were obtained in order to determine the D.P. of cellulose under a wide range of conditions, before and after treatments which may reduce the lengths of the molecules. Cotton fibers receive somewhat harsh treatments in the field before harvest, during processing, in laundering, and other treatments while in use. These studies were made in order to determine the magnitude of the variability in D.P. before treatment and the changes with simple treatments immediately after the bolls were opened and the fibers were exposed to light. The effects of chemical degradation as well as preharvest conditions in the field were studied. In order to determine the extent of these changes, samples were taken from unopened bolls which presumably would be undamaged and, therefore, contain fibers with a maximum D.P.

Additional studies are being made to determine the extent to which D.P. varies with variety and location of growth and its relationship with other physical properties of the fiber.

Materials and Methods

Cotton fibers from unopened bolls (grown at Beltsville, Md.) were used to determine D.P. at different stages of development. The bolls were separated into samples that contained (1) primary walls only and (2) those that contained secondary thickening in

This paper was presented at the Fall Meeting of the Fiber Society, Boston, September 9, 1948.

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addition to the primary wall. The fibers with primary walls were found to contain only 73.6 percent cellulose, whereas those with well-developed secondary walls (*i.e.*, nearing maturity) had as much as 95.1 percent cellulose. The method of sampling removed much of the sugar and other soluble fractions from the fresh, undried fibers, which accounts for the relatively high cellulose value, particularly in the samples composed entirely of primary walls.

To facilitate sampling, the fiber was removed from the seed and placed in water to form a slurry. Excess water from the sample was removed by pressing the fiber between paper towels. The portion of each sample used as a control was dried in alcohol and ether.

Fibers with primary walls only shrank into a hard mass when dried at 105°C or above, and were softened or swollen before nitrating. This was done by digesting with ethylene glycol for several hours at 80°C on a steam bath. Samples from mature cotton, such as that used for studies on the seed and lock, were allowed to stand overnight in alcohol. After the removal of the alcohol, the samples were boiled for 1 hour in 1.0-percent NaOH and ¼ hour in H_3BO_3. Cottons picked just as the bolls opened were used in the studies of fibers from various parts of the seed and boll in order to keep degradation at a minimum.

The solution of cellulose nitrate has many advantages in the determination of the degree of polymerization, in that cellulose degradation is held to a minimum and extraneous fiber materials other than cellulose are reasonably well eliminated. The use of

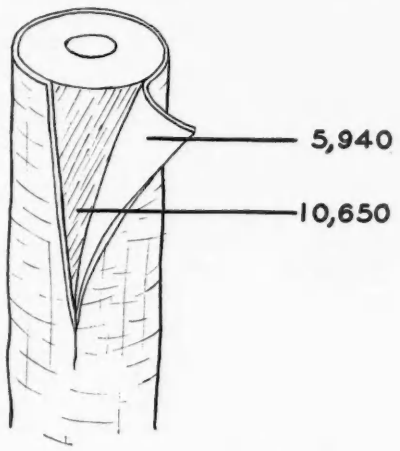

FIG. 1. *Diagram of cotton fiber showing the primary wall peeled back with a D.P. of 5,940 and the secondary wall beneath with a D.P. of 10,650.*

H_3PO_4 and P_2O_5 by Berl and Rueff [4] and Berl [5] as a dehydrating agent have hastened the time of nitration. Contact with the nitrating reagent after the reaction is completed, up to several hours, showed little evidence of degradation. Staudinger's [30] work with long-chain macromolecules has been the basis for much of the viscosity-molecular weight data of recent years. Kraemer [23] introduced the intrinsic viscosity function

$$\text{D.P.} = K\,[\eta],$$

where D.P. = degree of polymerization, $[\eta]$ is the intrinsic viscosity, and K = a constant,

$$\text{from} \qquad [\eta] = \left[\frac{\ln \eta r}{C}\right]_{\text{limits } C=0} = \left[\frac{\eta sp}{C}\right]_{\text{limits } C=0},$$

where C = concentration, ηr = relative viscosity, and ηsp = specific viscosity.

Coppick [12] was able to show that modification of the Farrow and Neal [14] equation $1 + K/C = B \log \eta r$, where K and B are constants, agreed very well with the intrinsic viscosity function of Kraemer [23].

The degree of polymerization was determined by nitrating cotton fiber and dissolving the cellulose nitrate in butyl acetate. From the viscosity of the solution the D.P. was calculated. The samples were nitrated for 2 hours at 10°C with a mixture of 64 percent HNO_3, 26 percent H_3PO_4, 10 percent P_2O_5, in a ratio of fiber to nitrating mixture of 1 to 100. The system was stabilized by the method described by Berl [5], in which the cellulose nitrate is immersed in 50-percent alcohol at − 10°C after the fiber is freed from the nitrating mixture. This is followed by boiling the nitrated fiber with three different portions of 95-percent alcohol for 5-minute intervals. After the final alcohol treatment, the samples were partially dried by suction and the residual alcohol was removed with ether. The solution of the cellulose nitrate in butyl acetate was made by shaking in small bottles a quantity sufficient to make a 0.1-percent solution. A Cannon and Fenske [11] viscosity pipette was used and the viscosity was determined at 25° ± 0.02°C. The time of flow was at least 100 seconds. The pipettes were calibrated with Bureau of Standards oils and from $\eta r = \frac{\eta}{\eta_0}$; the intrinsic viscosity was calculated from the equation of Farrow and Neal [14] as modified by Coppick [12]:

$$[\eta] = \frac{\ln \eta r}{C}(1 + 0.5\,C).$$

TABLE I. VARIATIONS IN DEGREE OF POLYMERIZATION OF CELLULOSE FROM COTTON FIBERS, WITH PRIMARY WALLS ONLY AND WITH SECONDARY THICKENING IN ADDITION TO THE PRIMARY WALLS, AS AFFECTED BY DIFFERENT METHODS OF DRYING

Treatment	D.P. of fibers with primary walls only (No. glucose anhydrides)	Difference from control (%)	D.P. of fibers with primary and secondary walls (No. glucose anhydrides)	Difference from control (%)
Control	5,940		10,650	
Air-dried at 20°C	3,142	−47.1	10,152	− 4.6
Oven-dried at 55°C for 18 hrs.	3,630	−38.9	10,880	2.2
Oven-dried at 105°C for 4 hrs.	2,100	−64.7	10,030	− 5.8
Oven-dried at 150°C for 4 hrs.	1,739	−70.8	7,488	−29.7
Dioxane at 105°C; boiled 2 hrs.	4,217	−29.0	7,480	−29.8
Ethylene glycol at 197°C; boiled 2 hrs.	4,370	−26.4	9,702	− 8.8
Glycerol at 290°C; boiled 2 hrs.	5,340	−10.1	9,900	− 7.0
Boiled 1 hr. in 1% NaOH and ¼ hr. in 4% H_3BO_3	6,208	4.5	10,880	2.2

This equation was found to give values which compare favorably with the results obtained by extrapolating values of $\frac{\ln \eta r}{C}$ at infinite dilution. From the intrinsic viscosity the degree of polymerization was calculated from D.P. $= K[\eta]$, where $K = 270$, from the work of Kraemer [23]. The magnitude of the D.P.'s reported here is determined by the formulas used; however, this does not affect the comparison of one sample with another.

The method described nitrates cotton very uniformly to approximately $13.0\% \pm 0.2\%$ nitrogen. The nitrogen in the cellulose nitrate was determined by the Lunge nitrometer.

Results

Cotton with only the primary wall was found to have a lower degree of polymerization and to be more sensitve to drying treatments than was mature cotton. Table I shows the degree of polymerization of cellulose from cotton fibers with primary walls only, and with secondary walls in addition, respectively, as influenced by dry heat and heat applied by boiling in solvents with different boiling ranges.

Cellulose in the primary wall was readily degraded in the presence of moisture at room temperature and above. The D.P. was numerically greater in fibers dried at 55°C than in those dried at room temperature. Although the data are limited, the differences observed may be significant, since both fibers with primary walls only and those with secondary walls in addition show the same trend—13 percent and 7 percent greater D.P.'s, respectively—and the viscosity determinations are accurate to about 5 percent. Further data are needed, however, to determine whether or not the slower drying permits more degradation.

At 105° to 150°C the effects of heat are pronounced. Heat from 105° to 290°C, when organic solvents are used to replace water, degrades the cellulose much less than when water alone is present. The degradation in solvents appears to follow molecular activity more than temperature, since glycerol, which boils at 290°C, gave the highest D.P.—that is, the least degradation. Boiling in a solution of 1-percent NaOH eliminates most of the noncellulosic materials without degradation of the residual cellulose of the fibers taken from unopened bolls.

Degradation due to sunlight, which may be accelerated by moisture and biological decay, tends to reduce the D.P.'s of cottons exposed in the field (Table II).

Both varieties reported in Table II show relatively high D.P.'s before exposure to the weather. The table shows the actual weathering with respect to sunshine, cloudiness, and rainfall for California. For New Mexico, these data were not available; however, during the exposure period little rain fell and the days were bright and sunny. The California samples showed a drop in D.P. of 18 percent in 43 days of sunshine, whereas the New Mexico samples lost 27 percent in 90 days' exposure to sunshine. The kier-boiled samples showed additional losses of 15 to 23 percent in D.P. for the samples from California and New Mexico, respectively. After kier-boiling the California samples lost a total of 37 percent and the New Mexico samples lost 41 percent of their original chain lengths on the basis of the unexposed samples. It will be observed, however, that the un-

TABLE II. EFFECTS OF WEATHERING ON THE DEGREE OF POLYMERIZATION OF CELLULOSE IN COTTON FIBERS

Variety	Location of growth	Weathering in the field		Exposure in field after bolls opened (Days)	Degree of polymerization	
		Sunshine (Days)	Cloudy (Days)		Without kier boil (No. glucose anhydrides)	With kier boil
Acala	Shafter, Cal.	None	None	0	10,388	8,860
Selection		43*	None	43	8,499	6,540
5-12-7-6		43	38†	81	8,247	7,085
Acala	State College, N. Mex.	None	None	0	10,320	8,512
W-29-1		90	None	90	7,540	6,140

* Rainfall 0.43 inch.
† Rainfall 2.27 inches.

exposed samples also lost in D.P. upon boiling in NaOH.

A study was also made of the degrees of polymerization at the different positions on the seed and in the lock. In general, the D.P. was greatest in the fibers from the region of the micropyle, or sharp, pointed end of the seed, and lowest in the fiber on the chalazal cap, or blunt end of the seed, as shown in Figure 2. The fibers on the chalazal cap were thin-walled and therefore contained a higher percentage of primary wall, which was found to contain shorter molecules (Table I) than the secondary wall.

Figure 3 gives the D.P.'s for different positions in the lock. The D.P.'s were lowest at the tip of the lock and became progressively higher towards the base.

Discussion

Studies of the structure and chemistry of cotton fibers from unopened bolls have been reported by other workers [3, 6, 13, 15, 24, 26, 28, and 31]. The literature is replete with degree-of-polymerization studies on the mature cotton fiber as found in commercial bales. Most of this work was done on fibers without a knowledge of their previous treatment. Since there is a gradual degradation of cotton fiber once it is exposed to the atmosphere, an accurate value for the original or undegraded chain lengths cannot be obtained from such material.

The data reported here show that the degree of polymerization of the cellulose in a given cotton fiber is dependent upon many factors, among which are: its development or cell-wall thickness, variety and location of growth, and degradation or damage. It is highly probable that enzymes are active in moist cellulose during the first drying when the bolls open, thus contributing to the lower degree of polymerization found when fibers were dried at room temperature. Elevated temperatures may stop enzymatic activity, but at 105°C and above there was a marked reduction in D.P. when water was present. When the water was replaced by solvents, the effect of the temperature was reduced. The degradation in solvents appears to follow molecular activity more than it does temperature, since glycerol, which boils at 290°C, gave the least reduction in D.P. It is also possible that traces of peroxides in the dioxane would degrade the cellulose.

The primary-walled cotton contained 73.6 percent cellulose and 25.4 percent of other materials which were not analyzed. The action of strong nitrating acid on these materials is open to speculation. Pectic substances, waxes, and proteins, if not removed, may be sufficient to interfere with viscosity and degree-of-polymerization determinations. The nitration work reported on pentosans indicates that hydrolysis may result and difficulty in nitration may be experienced when they are present. In such cases a small amount of nitrated pentosan material will be present in the solution [27, 29]. According to Hanglein [19], pectic substances are partially hydrolyzed by the strong nitrating acids and the residue dissolves in the nitrating mixture. Proteins would also be partially hydrolyzed and soluble in the strong acid. Therefore, the materials that do not dissolve in the nitrating solution and are not removed during stabilization will eventually appear as a suspension in the butyl acetate. This suspension was noticeable in some of the cellulose nitrate solutions from fibers with primary walls only; however, it could not be evaluated because of the small amount. It is possible that these suspensions may affect the viscosity results;

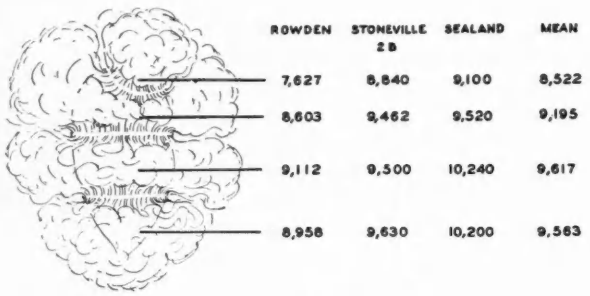

FIG. 2. *Outline of cotton seed and degree of polymerization of cellulose for three varieties of cotton.*

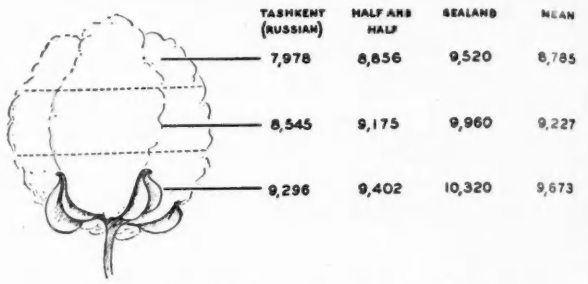

FIG. 3. *Outline of open ball and degree of polymerization of cellulose in three areas of the ball for three varieties of cotton.*

however, filtering the solutions through sintered glass showed no significant changes in the rate of flow of the solutions.

Preliminary studies indicate that most of the extraneous materials were eliminated during the nitration of 2 hours and during the subsequent stabilization; otherwise, a weighable amount of undissolved material probably would have been present in the butyl acetate solution.

Boiling undamaged cotton in 1.0-percent NaOH eliminates most of the extraneous material of the fiber [20], without appreciable degradation. There is little evidence that this treatment will remove any cellulose other than highly degraded molecules. By comparing the control value for degree of polymerization with the kier-boiled sample, an indication is obtained as to how well the nitration method eliminates the materials other than cellulose. The fact that the difference between control and kier-boiled samples is only 4.5 percent is evidence that most of these extraneous materials have been eliminated. A mild alkali boil has been used to determine the presence of modified bonds which are not severed in the acid nitration method. Damaged bonds from photochemical oxidation are not expected to be present in fiber from unopened bolls and no degradation was observed between the control and the kier-boiled samples. The increases in D.P. in the kier-boiled samples (Table I) are not considered to be significant and may be attributed to the removal of extraneous noncellulosic materials.

The degree of polymerization of the control sample with secondary wall is approximately 44 percent higher than that of fiber with primary wall only. The greatest difference in the two cottons is, however, their sensitivity to dry heat. Even drying at room temperature dropped the D.P. of primary-wall fiber by 38.9 percent, whereas the D.P. of cotton fiber with secondary wall decreased only 4.5 percent from that of the control. The primary-wall cotton was degraded at all temperatures, whereas the secondary-wall cotton was degraded substantially only at the 150°C temperature. When the water was replaced by solvents the greatest degradation of the cellulose chains resulted in both types when the cotton was boiled in dioxane. With the higher-boiling ethylene glycol and glycerol, loss of about 9 percent and 7 percent in D.P., respectively, was observed on the basis of the control. The fibers with primary wall lost only 20 percent and 10 percent in D.P., respectively, when the water was replaced by ethylene glycol and glycerol, which indicates a greater stability for the well-matured fibers.

The effect of sunlight on cotton was reported as early as 1883 [33] and the work of Bot and van Vreeswijk [10] demonstrated that cotton was somewhat sensitive to oxidation upon irradiation under certain conditions. While the action of light on fibers is not very well known, it is generally considered to be photochemical and the ultimate change requires oxygen in the presence of moisture. Some discussion may be in order on the effects of a kier boil on field-damaged cotton. In the determination of D.P. by the cellulose nitrate method, modified bonds are supposedly not broken. To get a true measure of the effect of light, the damaged oxygen bonds should be severed to determine the full extent of weathering. Even a mild alkaline treatment hydrolyzes oxycellulose, thus rupturing primary valency bonds that have been damaged. The data on unopened bolls in Table I showed that the alkali boil did not affect the D.P. when the fiber had not been exposed to light. The oxidation brought out by the kier boil from bolls only slightly exposed to light appears to be a photo-

chemical oxidation which is exceedingly rapid in the moist fiber as the bolls are opening. Actually, an initial loss in D.P. of 14.7 percent for Shafter and 17.5 percent for State College samples was found soon after the bolls opened. On exposure to light fiber from both locations lost 37.8 and 36.1 percent, respectively, in spite of a 47-day difference in exposure. This indicates a rapid chain reaction at first, which satisfies the more easily oxidizable bonds and finally levels off to a slower reaction rate [25].

The data in Table II show a reduction in D.P. with time of exposure to light and a further reduction when the fibers are boiled in NaOH. Bonds that were weakened but not severed by the exposure to light were broken by the alkali treatment.

The low D.P. value obtained in the fiber from the region of the chalazal cap of the seed is probably due to the higher percentage of primary walls in the thin-walled fiber from this area. There are differences in the D.P.'s from the three varieties represented in Figures 2 and 3. Further confirmation of this will be published later in a more extensive treatment of varietal differences. The variations over the seed and lock, although significant, are not so great as those resulting from degradation associated with field damage.

It has been suggested by Pacsu [25] that the cellulose chain is made up of regular repeating multiple units of 256 glucose anhydrides each. Consequently, in the degradation of cellulose the degree of polymerization would, in such a case, be in multiples of these units. Data on the degree of polymerization in this paper appear to be multiples of from 7 to 43 times the unit 256, although no specific study of this relationship has been made.

The values reported here for degree of polymerization are higher than those usually reported in the literature; however, more recent values reported by Gralén and Svedberg [18] and Gralén [17] from sedimentation and diffusion rates and later reviewed by Mark and associates [2] indicate that the old values are much too low. Gralén's [17] top value for raw cotton was 10,800. By the removal of oxygen from the histological canals of the fiber with helium in a special apparatus, Golova [16] was able to obtain D.P.'s as high as 10,000 by the cuprammonium method. More recently Pacsu [25] has postulated that "(*a*) native cellulose possesses an infinitely high molecular weight; (*b*) as a consequence of common procedure adhered to by most investigators, native cellulose is unintentionally degraded to about D.P. 3,000, which is then reported as the average molecular weight of cellulose."

Summary

The degree of polymerization of cotton fiber with primary walls only and with secondary walls gave average values of 5,940 and 10,650, respectively. A kier boil of 1 hour had very little effect on the D.P. of fiber that had not been exposed to light or other degradation. Cotton fiber, particularly that with primary walls only, was very sensitive to drying, whether at room temperature or by heat, and lost as much as 70.8 percent of its D.P. in drying for 4 hours at 150°C. Fibers dehydrated by boiling in organic solvents did not show so great a degradation as oven-dried ones, which emphasizes the role of water in the reactivity of cellulose exposed to oxygen. The secondary-walled fiber was much more resistant to depolymerization associated with drying and was quite resistant to degradation at temperatures below 150°C. In the solvent dehydration the greatest degradation was associated with solvents of lower boiling points. This was true for fiber with and without secondary thickening.

The fiber from the half of the seed near the micropyle, or sharp point of the seed, was fairly uniform and had a maximum D.P. which decreased toward the chalazal cap. Within the cotton boll the D.P. was highest at the base of the lock, with a progressive decrease from base to tip.

The weathering of cotton fiber due to sunlight was very pronounced and appeared to start as soon as the fiber was exposed. Degradation measured by degree of polymerization, from bolls at the time of opening and after 43 days of sunshine, was 37 percent, whereas 41 percent decrease was noted over a 90-day period of exposure to mostly sunshine in another area. It is indicated that the rate of change in D.P. decreases with time. This may be due to the failure of the short light waves to penetrate the mass of cotton, thus causing the greatest degradation on the surface of the fiber mass, or to a greater sensitivity of the extremely long molecules to ultraviolet light.

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(Manuscript received May 17, 1948.)

Discussion of Published Papers*

Remarks on the "Secondary Yield Point" in Wet Wool Fibers

Harris M. Burte†

Contribution of the Textile Research Institute and the Department of Chemical Engineering, Princeton University, Princeton, New Jersey

IN A RECENT ARTICLE,* the secondary yield point (see Figure 1), a feature of the stress-strain diagrams for several of the wool fibers tested, was described. Although the data did not then indicate the following, it now appears that the explanation of this phenomenon is not the molecular one given, but depends upon the macroscopic dimensions of the fiber.

The secondary yield point is due to variations of the cross-sectional area along the length of the fiber tested. This has been proved in two ways:

1. Careful microscopic measurement has shown that all fibers which show this phenomenon vary significantly in their cross-sectional areas along their length. The converse is also true.
2. If a fiber which shows the secondary yield point is subdivided into several short lengths (to eliminate non-uniformity of cross-sectional area in the specimen tested) the secondary yield point no longer appears.

In Figure 1 the force-elongation curve ‡ of a fiber which showed an appreciable taper is given. Also shown are the curves for sections from either end of this fiber. It is evident that if these two sections are connected in series, as they were in the original fiber, two yield points at forces corresponding roughly to A and B would appear.

Although the over-all theory presented in the original paper * for the mechanical behavior of wet wool fibers is not affected by these findings, there is no longer any basis for postulating a discontinuity in the spectrum of states existing in the folded configuration. Rather, the theory as developed for Type I fibers should be applied to all fibers after due correction has been made for variation of cross-sectional area along the length of the test specimen. Such a correction would obviously be very tedious and uniform fibers would be most profitably investigated.

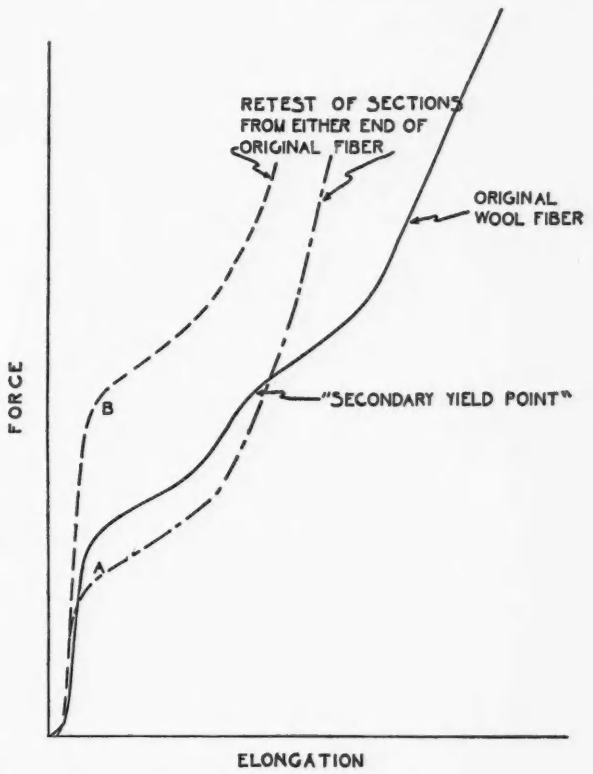

FIG. 1. *A stress-strain curve showing the secondary yield point and the stress-strain curves of segments from either end of the original fiber.*

* The paper discussed here is that by Burte, H., Halsey, G., and Dillon, J. H., "A New Concept of the Mechanical Behavior of Fibers," TEXTILE RESEARCH JOURNAL **18**, 449–69 (Aug. 1948). (The manuscript of these "Remarks" was received August 25, 1948, after the original paper had gone to press.)

† Fellow of the Textile Research Institute.

‡ This fiber was tested in water at a constant rate of extension.

ABSTRACTS

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A list of periodicals abstracted, the abbreviations used in references to articles, and the addresses of publishers of English language publications is given in the January, 1948, issue, page 48.

ANALYSIS: TESTING: LABORATORY METHODS

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Acetyl Determination

Quantitative estimation as acetic acid of acetyl, ethylidene, ethoxy, and α-hydroxyethyl groups. R. U. Lemieux and C. B. Purves. *Can. J. Research* **25B**, 485–9 (Sept. 1947).

Kuhn and L'Orsa's method for estimating terminal CH_3 and similar groups was based on the recovery of the acetic acid formed when such units were oxidized with hot concentrated chromic acid. The method has now been reduced to the semimicro scale and refined to the point were good analyses for acetyl, ethylidene, ethoxy, and α-hydroxyethyl groups in a variety of substances are obtained. Acetylated triphenylmethyl cellulose ethers, which do not seem amenable to customary acetyl analyses, give good results by the present procedure. Authors

Text. Research J. Oct. 1948

Conductivity Cell

Note on a conductivity cell usable with viscous liquids, pastes, suspensions, and wetted solids. Earl Otto. *Rev. Sci. Instruments* **19**, 368 (May 1948).

A new conductivity cell is described and illustrated which can be used to determine conductivity of viscous liquids, gels, pastes, emulsions, suspensions, and wetted powders as well as free-flowing liquids. The cell is easy to assemble and disassemble and usable over a wide range of temperature. The cell was designed primarily for use with highly conductive dispersions. Highest precision is not claimed but duplicate determinations deviate from the average by less than 0.2%. L. P. Witnauer

Text. Research J. Oct. 1948

Vacuum Properties of Dielectrics

Vacuum properties of synthetic dielectrics. Benjamin G. Hogg and Henry E. Duckworth. *Rev. Sci. Instruments* **19**, 331–2 (May 1948).

A method for determining a measure of the vapor pressure of 28 commercially available synthetic dielectrics is given and the results obtained reported. Wide differences in the vapor pressures were observed. Polystyrene, teflon, and mycalex possess a low vapor pressure and are particularly well adapted for use in vacuum systems. L. P. Witnauer

Text. Research J. Oct. 1948

Textile Fading Lamp Control

Textile fading lamps controlled with new light-sensitive papers. Anon. *Textile Age* **12**, 66, 68–70 (June 1948).

The development of a light-sensitive paper makes possible the standardization of tests to determine the light fastness of various fibers and dyes. The variation between different lights, or of one light over a period of time, may be corrected by using the light-sensitive paper to obtain a standard. A. L. Landau

Text. Research J. Oct. 1948

Microscopy

On interference microscopy. Sir Thomas Merton. *Proc. Roy. Soc. (London)* **A191**, 1–6 (Sept. 26, 1947).

An extension is described of the method discussed in *Proc. Roy. Soc. (London)* **A189**, 309 (1947), which makes it possible to apply interference microscopy to thicker specimens than was possible heretofore. W. E. Davis

Text. Research J. Oct. 1948

Identification of Processing Agents

Qualitative analysis of textile processing agents. H. B. Goldstein. *Am. Dyestuff Reptr.* **36**, 629–40 (Nov. 3, 1947).

A scheme of analysis is presented for the qualitative identification of textile processing agents. Included are cationic agents, delusterants, flameproofing agents, soaps, mercerizing agents, softeners, synthetic resins, synthetic surface-active agents, thickeners and sizes, and water-repellents. Methods and techniques are described in detail.
K. S. Campbell

Text. Research J. Oct. 1948

Measuring Stress Relaxation

An instrument for measuring stress relaxation of high polymer materials. W. S. Macdonald and A. Ushakoff. *J. Colloid Sci.* **3**, 135–45 (May 1948).

An apparatus for measuring stress relaxation consists of a measuring jig capable of applying a desired initial stress to the sample and measuring its relaxation continuously, and a suitable recorder. The application of this instrument using synthetic rubbers, nylon, leather, masonite, etc., for different measurements is described. E. D. Klug

Text. Research J. Oct. 1948

Modern Viscometers

The effect the consistency curve has on the development of modern viscometers. Henry Green. *J. Colloid Sci.* **3**, 129–33 (May 1948).

Consistency curves (stress vs. rate of shear) indicate that for a capillary viscometer the curve never becomes linear whereas for a rotating cup-type viscometer it does become linear. The latter is therefore more desirable since it permits determining yield values (intercept of the linear portion with the ordinate). From this the plastic viscosity may be calculated. The rotating cup viscometer is very desirable because rates of shear can be readily changed. Such viscometers with the high rates of shear corresponding to those in roller coating machines and printing presses remain to be developed. E. D. Klug

Text. Research J. Oct. 1948

Water in Bleaching Powder

Determination of water in bleaching powder. Jerome Goldenson and Charles E. Danner. *Anal. Chem.* **20**, 359–60 (Apr. 1948).

A distillation method is described for the determination of water in bleaching powder and high-test calcium hypochlorites which involves heating the sample with *o*-dichlorobenzene to drive all the water into the graduated separator tube of a specially constructed 1-piece apparatus. Author

Text. Research J. Oct. 1948

CHEMICAL AND PHYSICAL RESEARCH

★

Acetolysis of Cellulose

Hydro- and solvolytic and intramolecular oxidation-reduction. I. Acetolysis of cellulose as solvolytic oxidation-reduction. S. N. Danilov and P. T. Pastukhov. *J. Gen. Chem.* (U.S.S.R.) **17**, 1140–61 (1947) (in Russian) (through *Chem. Abstr.* **42**, 2499*d* (Apr. 20, 1948)).

Text. Research J. Oct. 1948

Swelling of Cellulose

Solvation and swelling of cellulosic polymers. P. Clément. *Ann. chim.* (12) **2**, 420–88 (1947) (through *Chem. Abstr.* **42**, 2765*c* (Apr. 20, 1948)).

Text. Research J. Oct. 1948

Chemistry of Cellulose Acetate

Cellulose acetate. XII. Ripening of cellulose acetate with $ZnCl_2$ as catalyst. XIII. Influence of ripening temperature on hydrolysis velocity. XIV. Relation between the composition of ripening bath and the hydrolysis velocity. Katsumoto Atsuki and Ikumi Kagawa. *J. Soc. Chem. Ind. Japan* **45**, 376–82 (1942); **XV. Ripening of cellulose acetate with sulfuric acid as catalyst. XVI. Velocity of hydrolysis and its mechanism. XVII. Depolymerization of cellulose acetate during ripening.** Katsumoto Atsuki, Ikumi Kagawa, Kiichi Okada, and Masao Wakui. *Ibid.* **45**, 434–43 (through *Chem. Abstr.* **42**, 3173*d* (May 10, 1948)).

Text. Research J. Oct. 1948

Cellulose Crystallinity

Crystallinity of hydrocellulose. F. C. Brenner, V. Frilette and H. Mark. *J. Am. Chem. Soc.* **70**, 877–8 (Feb. 1948).

The specific volume of the hydrocellulose formed by the acid hydrolysis of a beechwood pulp decreases with the time of the reaction and the percentage of crystallinity increases. The increase in crystallinity is greater than would be expected if it were due solely to the removal of the amorphous portion. This may indicate that with the rupture of a cellulose chain in an amorphous portion of the fiber, a process of crystallization is initiated.
A. R. Macormac

Text. Research J. Oct. 1948

Detergency Studies

Detergency Studies. William P. Utermohlen, Jr., and E. Louise Wallace. TEXTILE RESEARCH JOURNAL **17**, 670–88 (Dec. 1947).

A survey of the literature and the practice of detergency as applied to textiles has indicated that the manufacture of successful detergents has progressed further than has the fundamental science of detergency. Many of the basic factors in soiling of textile fibers and fabrics, and in cleaning of the soiled materials, are not yet fully recognized and satisfactorily understood. The work on detergency reported in these three papers was done at the Institute of Textile Technology. The first two present an initial study of the mechanisms of soiling of

cloth, and of the cleaning of soiled cloth, using mixed pigment-oil soils. By using a pigment whose presence can be accurately determined by chemical analysis, a definite improvement in the quantitative understanding of these processes has been made; this technique appears to be new. The washing of lightly and severely soiled samples has demonstrated that there is a qualitative as well as a quantitative difference between the two, and thus has contributed to the understanding of how solid soil clings to cloth. The third paper reports a study of the effect of various soiling mixtures and storage conditions upon the ease of washing of soiled cloth. It was found that soiled cloth becomes more difficult to wash after it has been stored for several weeks or months unless the oils in the mixtures used to soil the cloth are inert to the oxidizing action of air and unless the storage conditions are dry. Use of oils which undergo oxidative polymerization (particularly linseed and tung oils) was found to lead to permanent binding of pigment soil to cloth; this process appeared to be aided by the presence of moisture. The work reported in the three papers was done in order to establish a basis for comparative laboratory testing of detergents and washing processes and for other studies of practical value in textile detergency.

Text. Research J. Oct. 1948

Stresses and Birefringence in Rubber

Stresses and birefringence in rubber subjected to general homogeneous strain. L. R. G. Treloar. *Proc. Phys. Soc.* (*London*) **60**, 135–44 (Feb. 1948).

A study of the stresses and birefringence of dry and swollen rubber sheets subjected to two unequal strains in two perpendicular directions is reported. A square test sheet was used with 5 projecting lugs on each side for the application of the loads. The method eliminates the effect of the nonuniformity of strain in the vicinity of the edges of the sheet by making use of the principle of the guard plate. The dependence of the birefringence and of the principal stress difference on the extension ratios as predicted by the molecular network theory is shown. Examinations of each of the principal stresses separately reveal consistent deviations. Application of the general theory of Mooney appears to give an accurate representation of the properties of a swollen rubber, but is still inadequate for a dry rubber.

L. P. Witnauer

Text. Research J. Oct. 1948

Viscoelastic Properties of Paper

Paper as a viscoelastic body. III. Applicability of Eyring's and Halsey's theory to stress-strain diagram of paper. Bertil Ivarsson and Börje Steenberg. *Svensk Papperstidn.* **50**, 419–32 (1947) (in English) (through *Chem. Abstr.* **42**, 2768*d* (Apr. 20, 1948)); *cf.* TEXTILE RESEARCH JOURNAL **18**, 59 (Jan. 1948).

In continuation of the study (*C.A.* **41**, 6404*g*, 7115*h*) of paper as a viscoelastic body, the authors investigate the application to paper of Eyring's and Halsey's reaction-kinetic theory (*C.A.* **39**, 5495[9]) and the theory by Leaderman (*C.A.* **38**, 251[3]) on elastic and creep properties of filamentous materials. After explaining the 3-element mechanical model used by Eyring, a recapitulation is given of the mathematical basis for the theory and it is extended to cover the special case when it is applied to paper. Tables are given with illustration by an example for graphically obtaining the parameter values in the equations. Eyring's 3-element model is not sufficient to explain the stress-strain relationship in paper, since in this case there also enters an irreversible flow, termed microcreep. The outward and inward flow are not identical, owing to thixotropy. Paper is found to have frozen-in stress, the amount of which varies for different types of paper. Such stress is found highest for paper having high ratio of dynamic stiffness in the machine direction and in cross direction. Frozen-in stress is almost 0 for laboratory handsheets. With a simple trial and error method, the estimation of the frozen-in stress is demonstrated. Study of such stress in paper will probably throw light on the cause of anisotropic changes in dimensions of paper upon conditioning. References.

Text. Research J. Oct. 1948

Jute

The hemicelluloses of jute fiber. P. B. Sarkar, A. K. Mazumdar, and K. B. Pal. *J. Text. Inst.* **39**, T44–58 (Feb. 1948).

Extensive data are reported on the analysis of jute and of its incrusting substances, the so-called hemicelluloses. Conclusions based upon this data are: Jute cellulose, isolated by any of the standard methods, invariably contains some polyuronides. The holocellulose, prepared by the sodium chlorite method, can be made free from xylan by treatment with 9.3% NaOH at room temperature. The hexosans can be removed by cold dilute NaOH much more easily than the other constituents, and the wet strength of the fiber is adversely affected thereby. The polyuronides are not free but appear to be linked with pentosans. All of the hemicelluloses cannot be removed by treatment with NaOH, but removal of lignin facilitates the process. Cold NaOH removes none of the lignin, whereas boiling NaOH removes about 20%. Mixed units of xylose and glucose probably do not occur in the α-cellulose of jute fiber. Pentosans and hexosans are present in almost equal proportions. Loss of weight of holocellulose prepared from jute is maximum at 9.3% NaOH. Loss of weight of defatted jute in NaOH is the same in solutions ranging from 9.3 to 17.5%. No correlation is apparent between xylan content and quality ratio of jute fiber. Removal of a portion of the hemicelluloses (the hexosans mainly) from jute increases fiber flexibility. Contrary to Norman, jute hemicellulose does reduce alkaline copper solution. No correlation, however, was found between copper number and quality. There

are indications that the hemicelluloses act as cementing materials for the ultimate cells of the fiber. 28 references. A. R. Martin

Text. Research J. Oct. 1948

Viscosity of Polystyrene

Viscous flow of molten polystyrene. R. S. Spencer and R. E. Dillon. *J. Colloid Sci.* **3**, 163–79 (May 1948).

The non-Newtonian capillary flow of a polystyrene of molecular weight 360,000 was studied in the range 165 to 250°C and 0.2×10^{-5} to 30×10^{-5} dyne-cm.$^{-2}$ shearing stress. An empirical equation for the rate of shear-shearing stress curve is given. The limiting value of the viscosity as the shearing stress approached zero was calculated. The temperature dependence of rate of shear-shearing stress relationship was due to the variation of the zero-shear viscosity with temperature. Molecular orientation was observed and a method was determined for determining the relationship between highly elastic shearing strain and shearing stress from measurements of flow orientation and flow rates.

Text. Research J. Oct. 1948 E. D. Klug

Rheology of thermoplastic materials. I. Polystyrene. Rolf Buchdahl. *J. Colloid Sci.* **3**, 87–98 (May 1948).

Using a modified Stormer viscometer, curves are given for the deformation in number of revolutions with time at fixed shearing forces at 150, 190, and 220°C. At 220°C, the rate of shear is constant; below this it increases with time. The effect of resting periods is to temporarily decrease the rate of shear, the extent of the decrease depending on the time. Resting periods at room temperature had practically no effect on the deformation curve. Increasing the temperature during the rest period and returning to the original temperature decreased the rate of shear. Increasing the shearing force temporarily decreases the shearing rate at the lower shearing force for a short period of time. Data are given showing that the angular recovery after one revolution increases with shearing forces or speed of deformation. Data on the relationship between shearing force and rate of shear are also given. The results are interpreted in terms of molecular structure. E. D. Klug

Text. Research J. Oct. 1948

BLEACHING: DYEING: FINISHING

★

Detergents

New concepts and trends in the detergent field. Anthony M. Schwartz. *Soap Sanit. Chemicals* **24**, 51–3, 181, 183 (Apr. 1948).

A review.

Text. Research J. Oct. 1948

Nonionic Detergents

Nonionic detergents. George E. Barker. *Soap Sanit. Chemicals* **24**, 46–8, 65 (June 1948).

Data illustrative of the increase in detergency accomplished by adding nonionic detergents to solutions of anionic detergents and formulas for special applications of nonionic detergents are given. 4 references. A. R. Martin

Text. Research J. Oct. 1948

Use for Desizing Waste

Utilization of desizing washings for the culture of industrially important microorganisms. B. S. Lulla. *Current Sci.* (India) **17**, 24–5 (1948) (through *Chem. Abstr.* **42**, 3183*e* (May 10, 1948)).

Amylase solutions which have been used for desizing of textiles are good culture media for *Bacillus subtilis* (in view of further production of amylase) or for *Actinomyces griseus* for streptomycin production.

Text. Research J. Oct. 1948

Oxycellulose Defects

Oxycellulose defects can be avoided or cured. J. McCartney. *Textile World* **98**, 116, 214, 216, 218 (July 1948).

The formation of oxycellulose, or the oxidation of cellulose fibers, may be caused by many factors occurring during bleaching and mercerizing, and will affect the dyeing properties of the fibers. Tests for the presence of oxycellulose are given.

A. L. Landau

Text. Research J. Oct. 1948

Direct Dyes

Second report of the committee appointed by council to discuss the dyeing properties of direct cotton dyes. *J. Soc. Dyers and Colourists.* **64**, 145–6 (Apr. 1948).

The first report issued Sept., 1946, concerned cotton materials only. The second report shows that although the results vary from fiber to fiber of other cellulosic materials the tests are still applicable. Three viscose rayons, cuprammonium rayon, mercerized cotton, and linen were tested. One table of results is given on the viscose rayons.

J. A. Woodruff

Text. Research J. Oct. 1948

Fluorescent Dyes

Selection and use of fluorescent dyes on textile materials. R. Mansell. *Textile Age* **12**, 7, 10, 11, 14, 18, 20, 24 (June 1948).

A discussion of the uses and methods of dyeing textile fibers with fluorescent dyes, and the types of dyes most suitable. The application of such dyes to cotton, wool, nylon, and rayon is explained.

A. L. Landau

Text. Research J. Oct. 1948

Dyeing Synthetic Hosiery

Synthetics major problem in hosiery dyeing. V. T. Hartquist. *Textile World* **98**, 127, 201, 202 (July 1948).

Important dye-application properties of synthetic fibers are salt sensitivity, temperature of maximum affinity, migration rates, and rate of exhaustion. Filament fibers are stated to be less troublesome than

the staple form. The low coefficient of friction of nylon is responsible for the high percentage of pulled threads. A. L. Landau

Text. Research J. Oct. 1948

FIBERS: YARNS: FABRICS: MECHANICAL PROCESSES

★

Yarn Conditioning

Worsted yarn conditioning practice. R. S. Audley. *Fibres* **9**, 213–16 (June 1948).

The technical aspects of worsted yarn conditioning are covered. Different types of conditioning apparatus and operational details are described and illustrated. L. A. Fiori

Text. Research J. Oct. 1948

High-Speed Drying

New high-speed drying unit for slashers. Anon. *Rayon Text. Mo.* **29**, 68–9 (Apr. 1948).

A radically different, high-speed dry box for slashers, capable of processing at least 1,000 lbs. per hr. and of handling efficiently a wide range of yarns including 100% cotton, 100% wool, rayons, and blends of all kinds, has been developed. The principle involved, the operation of the dry box, the special apparatus such as the wet-splitting device, and the slasher specifications are described. L. A. Fiori

Text. Research J. Oct. 1948

Oiling Cotton

Oiling of cotton. H. Paton. *Textile Weekly* **41**, 480, 482, 484 (Mar. 12, 1948); *cf.* TEXTILE RESEARCH JOURNAL **18**, 192 (Mar. 1948).

Progress is reported in the oiling of cotton for yarns up to about 40s for both the spinners of raw cotton and waste mills using hard and soft waste. Among the advantages of this practice is a reduction of "fly" and dust in the card room. The aspects for successful application are: (1) the limits of oil addition to cotton are narrow, 0.1%–2%; (2) specially designed atomizing nozzles working on direct liquid pressure and free from "drip" when pressure is cut off must be used; (3) the spray must be so directed that none condenses on the hard surfaces of the machine; otherwise oil-saturated cotton will be carried forward; (4) the oil should be applied to the cotton as an emulsion; proportions ranging from 2 parts of water and 1 part of oil, to 6 parts of water and 1 part of oil give good results; (5) the oil must be applied as soon as the cotton becomes loose, preferably at the hopper of the bale breaker. Applying emulsion at the lap is not recommended. In opening and picking, oiling produced the following effects: cleaner atmosphere, cleaner machines, more compact droppings, and no less efficient cleaning of the cotton. At the drawing frames a greater amount of sliver can be put in the cans, which increases production; also, there are fewer end breaks and static is virtually eliminated. Roving-frame bobbins are more compact and larger packages can be made. In general, yarn strength seems to be increased; one mill reported that reducing the twist slightly gave a stronger yarn. In weaving there have been no adverse effects and some benefits: a fuller cloth and cops that do not fly off the shuttle tongue during hot weather. It is doubtful whether such a small quantity of oil could produce a visible deepening of shade—a tendency attributed to oil present in the dye bath. There are no visible effects in bleaching. In spinning rayon staple, the improvement in spinning justifies oiling. L. A. Fiori

Text. Research J. Oct. 1948

Predetermining Cotton Spinning Characteristics

Predetermining the spinning characteristics of cotton. J. F. Bogdan. *Textile Bulletin* **74**, 65, 66, 68 (June 1948).

The 3 main factors affecting yarn strength are fiber strength, fiber cohesion, and yarn evenness. Methods of testing these factors are described. A. L. Landau

Text. Research J. Oct. 1948

Tear Strength of Fabrics

Cloths from fibers. Gyorgy Gara. *Kém. Lapja* **4**, 139–41 (1943) (through *Chem. Abstr.* **42**, 2777*b* (Apr. 20, 1948)).

Tests were made on rayon cloth unfinished, treated with waterproof finish, treated with phenoplast finish, and treated with both. The wet tearing strength of unfinished cloth was 21 kg.; cloths treated with phenoplast showed values of 40 and 39 kg.; those treated with waterproof agents 41 and 33 kg.; those treated with both types of agents 47 and 45.5 kg.

Text. Research J. Oct. 1948

Effects of Twist

Ascertaining twist variations in two-fold worsted yarns. Foster Pickles. *Textile Recorder* **65**, 49–51 (Apr. 1948).

Discussed in detail is a method for determining whether warp stripiness in cloth is due to twist irregularity or to some other cause. A range of 6 counts (2/16s, 2/24s, 2/36s, 2/48s, 2/60s, and 2/70s) was tested for twist on lengths of 10 in. and also 1 in. Statistically, the 1-in. tests showed a greater variation than the 10-in. tests, as might be expected. Also, there is a progressive variation as turns per in. are increased, due apparently to increased twist and fineness of the yarn counts. Analysis of the yarn from the cloth showing warp stripiness and comparison with a standard yarn will determine whether the yarn is the cause of the defect. L. A. Fiori

Text. Research J. Oct. 1948

Wear Resistance

Fibre properties and wear resistance of garments. Anon. *Text. Mercury and Argus* **118**, 828–30 (June 4, 1948).

Tensile strength is no longer considered to be intimately connected with the durability or resistance of a textile fiber or manufactured material, e.g., a wool yarn which has been rendered shrinkproof through chemical treatment. It has a higher strength because of the adhesion of

the fibers to each other and a lower wear value because the fibers break more easily owing to their increased brittleness. Attention is drawn to the governing factors correlated with wear value of different types of knitted goods. L. A. Fiori

Text. Research J. Oct. 1948

Eliminating Overbeating

Proper loom adjustments eliminate "bird's eyes." A. Frantz. *Textile World* **98**, 110–11 (July 1948).

Improper adjustment of the warp tension and incorrect placement of the whiproll of the loom may be the cause of defective weaving. Correction of these faults is discussed. A. L. Landau

Text. Research J. Oct. 1948

Worsted Spinning

New worsted spinning system. Anon. *Text. Mercury and Argus* **118**, 587 (Apr. 23, 1948).

A new system of worsted-yarn production, requiring only one type of spinning machine and designed to replace the cap, ring, flyer, and mule types, has been developed on the centrifugal principle. Its application to cotton-yarn production is being investigated. L. A. Fiori

Text. Research J. Oct. 1948

Efficiency in Weaving and Slashing

Efficiency in weaving and slashing. Anon. *Am. Wool and Cotton Reptr.* **13**, 23, 25 (May 6, 1948).

A discussion of the types of slashers used for cotton, worsted, and synthetic fibers, together with the factors in slashing which affect the weaving performance. A. L. Landau

Text. Research J. Oct. 1948

Glass Laminates

Laminated materials of artificial resin with glass fibers. F. Bollenrath. *Kunststoffe ver. Kunststoff-Tech. u.-Anwend.* **36**, 73–80 (1946) (through *Chem. Abstr.* **42**, 2469*a* (Apr. 10, 1948)).

Layers of woven glass silk (fiber diameter up to 7 μ) were pressed together with phenol resins to form sheets and other shapes which later were pressed in forms to any desired shape. Very thorough tests are described and the results given in tables. The material is slightly hygroscopic, can stand high temperatures, has a small linear expansion coefficient, $9 - 12 \times 10^{-6}$ mm./mm./°C., an elasticity modulus of 3780 kg./sq. mm., tensile strength up to 80 kg./sq. mm., impact bending energy up to 264 cm.-kg./sq. cm. The phenol resin content of the finished material is about 31% of the finished weight and the specific changes from 1.90 to 2.11, according to the ratio of glass to resin. Some practical applications are given: handles, levers, etc.

Text. Research J. Oct. 1948

Processing Nylon Staple

Processing nylon staple on woolen machinery. R. D. Bennet. *Can. Text. J.* **65**, 46, 48, 50 (June 25, 1948).

Data on types of nylon suitable for blending with wool, blending technique, the machine sequence, details of card settings and speeds, spinning notes, and general observations on the production of a good quality blended yarn. L. A. Fiori

Text. Research J. Oct. 1948

Socks from Nylon Staple

Socks from nylon staple. Anon. *Rayon Text. Mo.* **29**, 80–1 (Apr. 1948).

This discussion deals primarily with socks produced from 100% nylon staple and from nylon-wool blends. Data indicate that at least 65% nylon should be used if a shrink-resistance treatment has not been given to the wool. With 65% nylon and proper mixing of the fibers before carding, the shrinkage will be significant. The abrasion resistance of socks increases as the % of nylon in the yarn mixture is increased. Consumer tests have shown that spun nylon socks which are properly knitted and finished are comfortable in all seasons and have a good appearance and hand. The pilling of nylon socks is sometimes more noticeable than that of wool, presumably because woolen pills, being weaker, wear off, whereas the nylon pills remain. Lightweight "wool-type" half hose of spun nylon are strong, abrasion-resistant, and stable to washing. Since nylon yarns differ in size from wool or worsted yarns of the same count, knitters must experiment to determine the proper nylon yarn for the fabric desired. Tables give data on yarn counts, deniers, proper blends of deniers for different types of socks and machines, knitting tension, dyeing, and finishing techniques. L. A. Fiori

Text. Research J. Oct. 1948

Upholstery Cloth

A new synthetic upholstery cloth. Anon. *Textile Weekly* **41**, 742 (Apr. 16, 1948); *Text. Mercury and Argus* **118**, 560 (Apr. 16, 1948).

A new synthetic woven material, "Tygan," with numerous potential uses and a particular appeal to upholsterers, is claimed to have the wearing qualities of leather, yet, like a fabric, can be woven in a variety of designs (twill, herringbone, etc.). Tygan is woven on specially equipped looms from extruded monofilaments of "Bexan," whose physical properties can be varied to suit specific end uses. Bexan can be manufactured in the colors of the British standard color range; the colors will be fast. Tygan is stated to be resistant to all kinds of weather, including tropical conditions. It reflects heat and is therefore warm in winter and cool in summer. L. A. Fiori

Text. Research J. Oct. 1948

Washable Wool

Washable wool interest peaks. Anon. *Am. Wool and Cotton Reptr.* **13**, 11, 12 (Mar. 25, 1948).

Continued progress in the manufacture of washable woolens and the approach to perfection of technique are evident as various companies prepare to offer materials of this type to the consumer. Shrink-

age of the woolen fabric after washing is not completely halted but is held to a very low percentage. Care must be taken in laundering even though these fabrics have been treated for shrinkage. A. L. Landau

Text. Research J. Oct. 1948

MISCELLANEOUS

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Abstracting

Abstracting research reports. L. C. Stork and K. C. Cousins. *J. Chem. Education* **25**, 130–3 (Mar. 1948).

Details on the preparation, classification, reproduction, and distribution of abstracts by the special abstracting service of the Texas Company, Beacon, New York.

R. K. Worner

Text. Research J. Oct. 1948

Industrial Adhesives

Industrial adhesives. Kenneth Rose. *Materials & Methods* **27**, No. 2, 94–104 (1948) (through *Chem. Abstr.* **42**, 2814*h* (Apr. 20, 1948))

Characteristics and uses are given for adhesives of natural or synthetic rubber, thermoplastic resins, thermosetting resins, resin-rubber compounds, and adhesives of animal, fish, and vegetable origin.

Text. Research J. Oct. 1948

Clothing Design

Functional clothes design. Clarice L. Scott, *J. Home Economics* **48**, 123–4 (Mar. 1948).

Research on design of work dresses in Bureau of Human Nutrition and Home Economics is discussed.

R. B. Evans

Text. Research J. Oct. 1948

Latex Patents

Latex patents. W. H. Stevens. *Trans. Inst. Rubber Ind.* **23**, 118–22 (1947) (through *Chem. Abstr.* **42**, 2796*f* (Apr. 20, 1948)).

A discussion of the increase in the number of British patents on natural-rubber latex from 1920 to 1939. A list of 55 patents which are regarded as outstanding is included.

Text. Research J. Oct. 1948

New Kellog Loom

New loom features unit system. Anon. *Textile Inds.* **112**, 102–4 (Apr. 1948).

The new Kellog loom retains the conventional weaving motions but is built with each basic motion as an intregal unit in itself. Recommended operating speeds range from 220 to 230 picks per min. and doffing may be done with the loom in motion. Descriptions of the unit sections are given. A. L. Landau

Text. Research J. Oct. 1948

Plastics

Major advances in plastics during 1947. James W. Underwood. *India Rubber World* **117**, 621–3 (1948) (through *Chem. Abstr.* **42**, 2812*i* (Apr. 20, 1948)).

A review, with 72 references.

Text. Research J. Oct. 1948

Polyamides in Germany

Polyamides in Germany. Anon. *Modern Plastics* **25**, 125–6, 172–7 (Mar. 1948); 148–50, 216, 218, 220, 222–4, 226–7 (Apr. 1948).

A digest and review of the many O.P.B. reports on this subject.

Text. Research J. Oct. 1948

Power Factors

Power factor and its improvement. Anon. *Textile Manufacturer* **74**, 224–7 (May 1948).

An explanation of the term "power factor" and the means of obtaining a higher factor. The relationship of voltage, current, and power with respect to different currents are shown. A. L. Landau

Text. Research J. Oct. 1948

Synthetic Protein Textiles

Artificial materials based on protein. Th. Koch. *Chem. Weekblad* **44**, 29–35 (1948) (through *Chem. Abstr.* **42**, 2436*h* (Apr. 10, 1948)).

A review, principally on synthetic textiles, with 48 references.

Text. Research J. Oct. 1948

Spinning Ring Wear

Wear in spinning rings. Anon. *Platts Bulletin* **6**, 35–7 (Mar.–Apr. 1948).

The metallurgist's problems concerning wear in spinning rings are presented. Photomicrographs illustrate the features mentioned.

Text. Research J. Oct. 1948 L. A. Fiori

Water Quality

Water quality essential to textile processes. V. J. Calise. *Textile World* **98**, 101–3 (July 1948).

A report of the impurities present in water and the defects which are caused by them in bleaching, mercerizing, and dyeing cotton, rayon, and wool. A. L. Landau

Text. Research J. Oct. 1948

Belgian Woolen Spinning Mill

Woolen spinning mill is Belgian equipped. G. Zellnik. *Textile World* **98**, 104, 105, 204, 206, 208, 210 (July 1948).

Description of Belgian-made equipment installed in an American plant. Distinctive features are two sets of interchangeable strippers for the picker, 78-$\frac{3}{4}$ in. card cylinders operating at 130 r.p.m., and tape condensers delivering 160 to 184 ends.

A. L. Landau

Text. Research J. Oct. 1948

Worsted Frame

Worsted frame is "universal spinner." C. H. Harrigan. *Text. Inds.* **112**, 79, 81 (May 1948).

Description of a worsted ring spinning frame claimed to be capable of spinning very fine counts. Drafts of a maximum of 20 may be attained by a drafting system of 6 lines of rolls. High spindle speeds are possible owing to the use of travelers containing beryllium copper.

A. L. Landau

Text. Research J. Oct. 1948